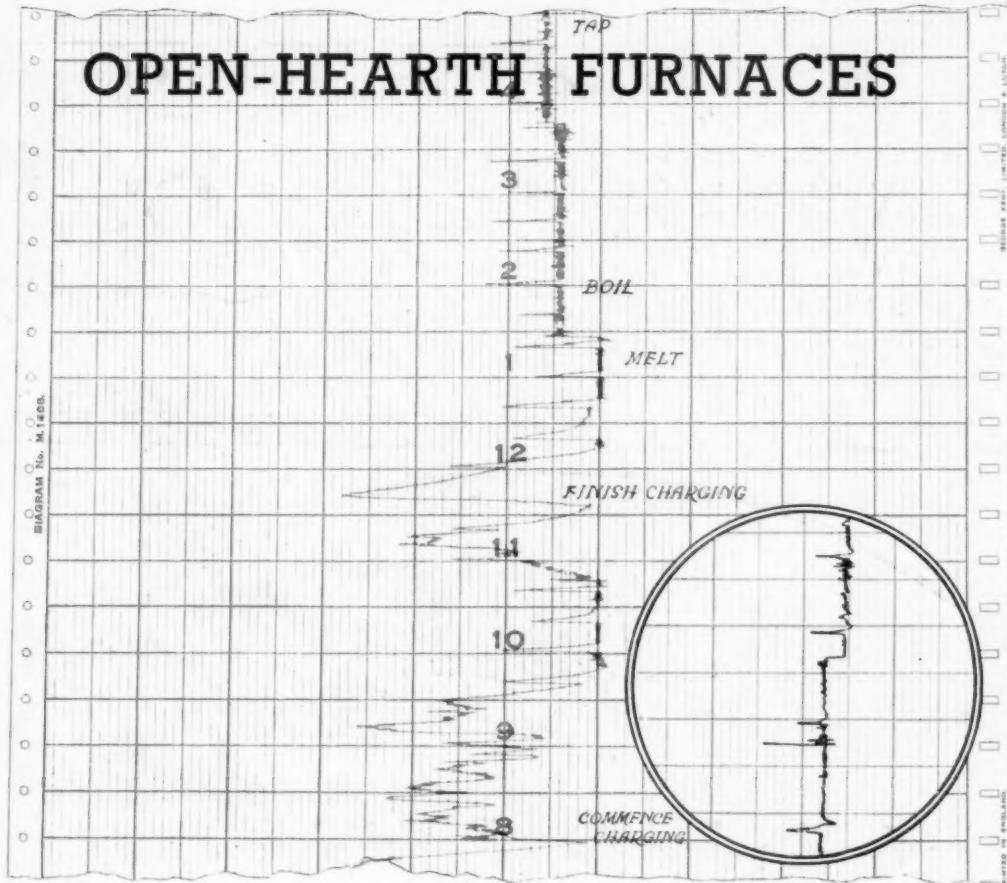


METALLURGIA

THE BRITISH JOURNAL OF METALS



Maintenance of optimum temperature

Without precision temperature control, the attainment of the highest melting efficiency is prohibited by the need for restricting the furnace temperature to a figure well below the optimum, in order that peak fluctuations may not damage the roof lining. George Kent Ltd., can now supply an automatic control system for maintaining the roof temperature between reversals within close limits.

These portions of a chart show the results being obtained on a furnace fired by hot raw producer gas by Messrs. John Baker & Bessemer Ltd. The steep drops in temperature are due to furnace reversals. The chart is 10" wide and one division of the furnace reversals corresponds approximately to 6°C. and the target temperature is maintained to the extremely close limits

despite variations in the furnace conditions, thereby conducting to considerably increased output. In order to show the stability of control, the chart was experimentally run at three times the normal speed; a portion of this is shown in the circle.

The Kent system, utilizing a standard Multelec, with the Mark 20 controller, is based on the latest automatic control practice and is available for oil or gas fired furnaces.

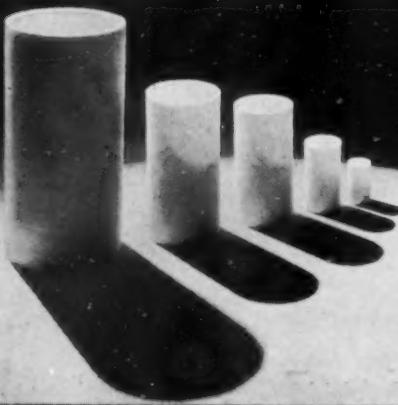
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GEORGE KENT

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We can now supply very highly refractory crucibles suitable for melting precious metals and for work at high temperatures, up to 1800° C., particularly where contamination due to silica must be reduced to a minimum. These FUSED ALUMINA and FUSED MAGNESIA crucibles are of the highest purity and without added siliceous bond. The largest vessel illustrated is 12 inches x 6 inches diameter.

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THE old saying is carried much further in manufacturing Rockweld electrodes. Not only are any two electrodes in our production guaranteed to be the same, but *any* two electrodes in *any* production are the same. Uniform quality is a characteristic of Rockweld production which ensures on every production job a perfect uniformity of performance.

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Useful Molybdenum Steels

Data Sheet 2

SPECIFICATION Nos. TENSILE STRENGTHS 40-100 tons/sq.in.

MINIMUM IZOD VALUES AND RULING SECTIONS—
FOR HARDENING & TEMPERING

Tensile Minimum Tons/Sq. In.	RULING SECTIONS IN INCHES							
	Up to 1 $\frac{1}{8}$ "		Up to 2 $\frac{1}{2}$ "		Up to 4"		Up to 6"	
B.S. En No.	Izod	En. No.	Izod	En. No.	Izod	En. No.	Izod	
40 tons						13	40	
45						16	40	
						17	40	
						19	40	
						29	40	
						100	40	
						110	40	
						160	40	
50				16	40	17	40	
				19	40	24	40	
				100	40	29	40	
				160	40	110	40	
55		16	40	17	40	24	40	
		19	40	110	40	25	40	
		100	40			27	40	
		160	40			29	40	
60	16	35	17	35	24	35	25	35
	100	35	19	35			26	35
	160	35	110	35			27	35
							28	35
							29	35
65	17	35	24	35	29	35	25	35
	19	35					26	35
	100	35					27	35
	110	35					28	35
70	19	30			25	30	26	30
	24	30			27	30		
	110	30			28	30		
					29	30		
75/85 and 80/90	24	25	25	25			26	25
		28	25					
100	24	8	25	10	26	10	30B†	15
			29	10				
			30B*	15				

* Air Hardened
† Oil Hardened

CLIMAX MOLYBDENUM COMPANY
OF EUROPE LIMITED

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FOR OUTSTANDING

STRENGTH
TOUGHNESS
ENDURANCE

Specify
**MOLYBDENUM
STEELS**

The presence of Molybdenum gives steel the ability to harden to great depth. Because of this effect, alloy steels containing Molybdenum can be heat-treated to develop the high combinations of strength, toughness and endurance required by the modern designer.

Remember also that of all the elements which markedly increase the hardenability of steel, Molybdenum is the only one which is not lost by oxidation from the molten bath. Consequently the hardenability imparted by Molybdenum can be closely controlled.

Specifiers of Molybdenum steels are thus assured of good, uniform response to heat-treatment.

MOLY



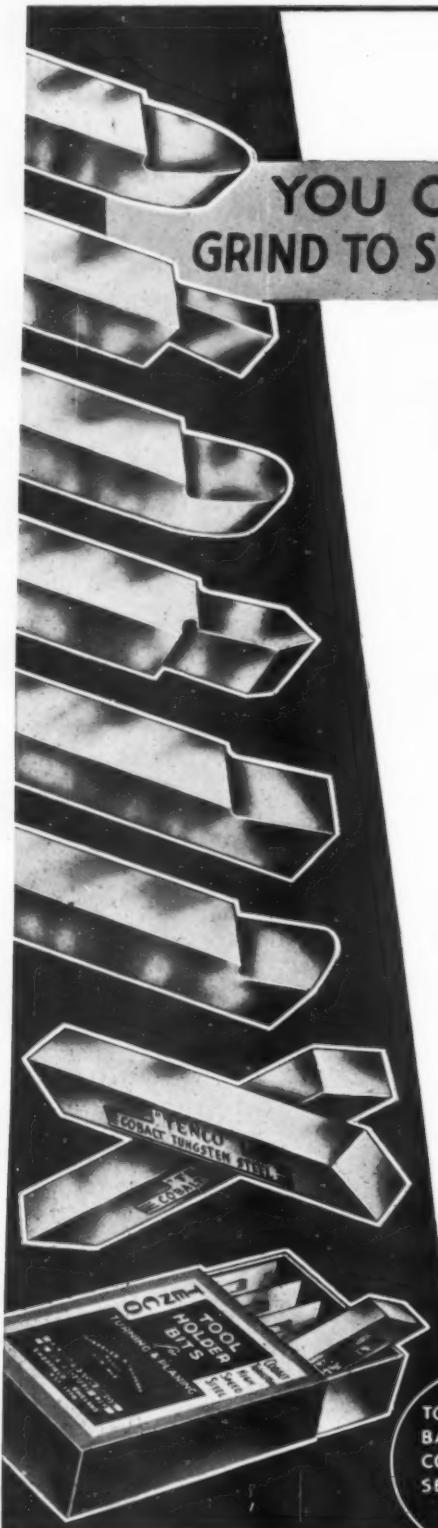
Every year there's less and less *g* at the B.I.F. and more and more lightness with strength. This is largely due to the activities of H.D.A., who during the last quarter of a century have spent an active life in reducing the power of *g* (for gravity) and taking a lot of excess weight out of industry. By using Hiduminium, Britain makes her wares attractive, light and strong. At Stand D.232 Castle Bromwich, Birmingham, there is ample evidence that plenty of opportunities still exist for people who choose to

... make light work of *g* with

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- ★ EACH TOOL-BIT TESTED AND GUARANTEED TO A STANDARD DEGREE OF HARDNESS

"TENCO" TOOL BITS ARE MADE IN STANDARD LENGTHS AND FROM $\frac{3}{16}$ " TO 1" SQUARE

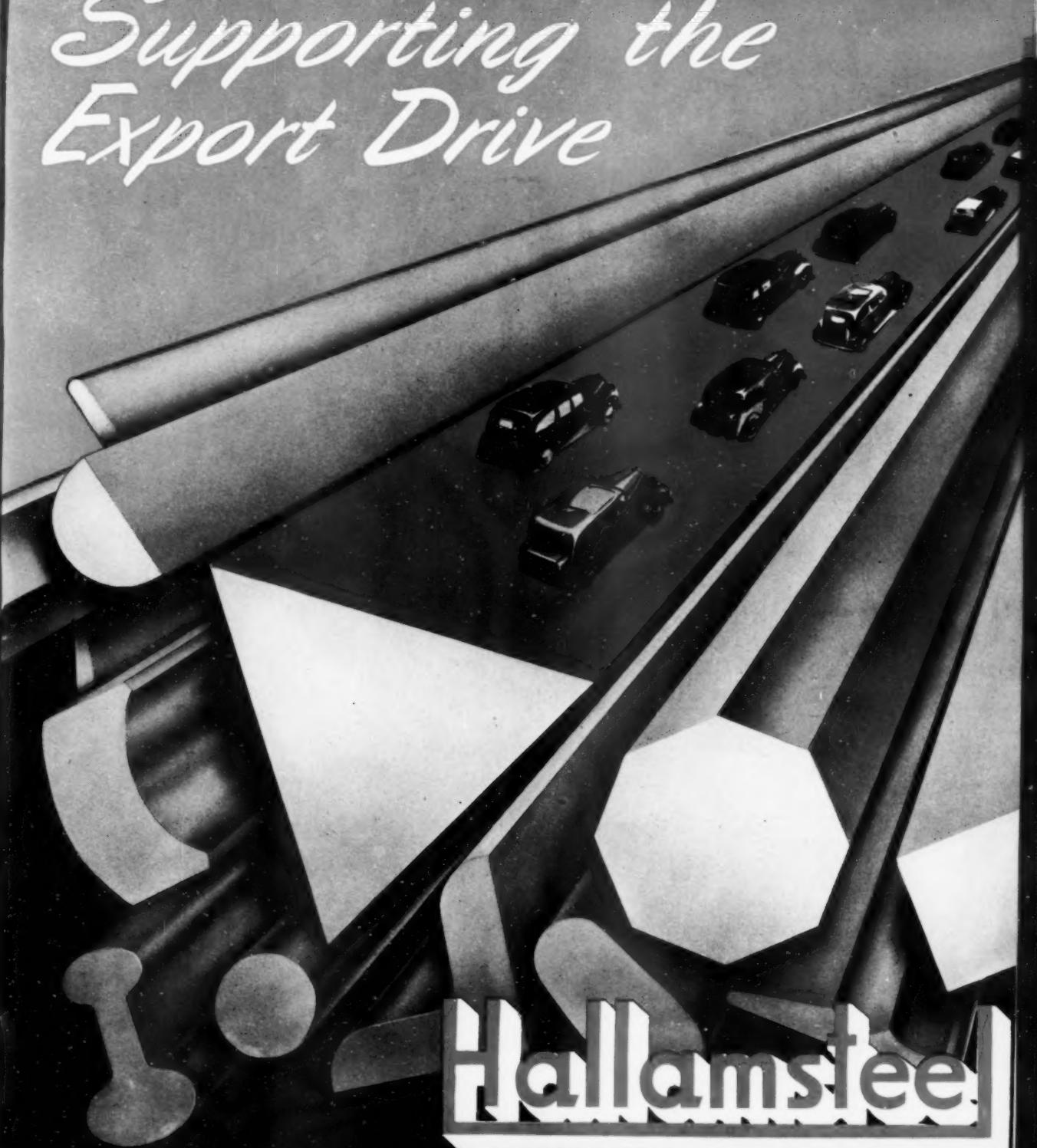
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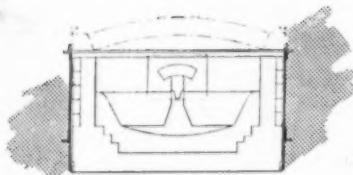
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Telephone : Sheffield 24304 (7 lines).

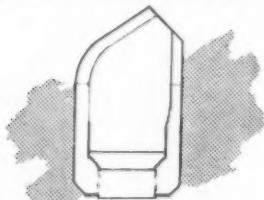
Telegrams : Hallamsteel, Sheffield

BRITAIN'S BEST BASIC REFRactories

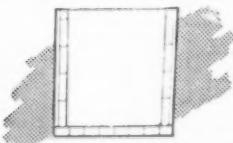
**G.R.
'341'
DOLOMITE BRICK**



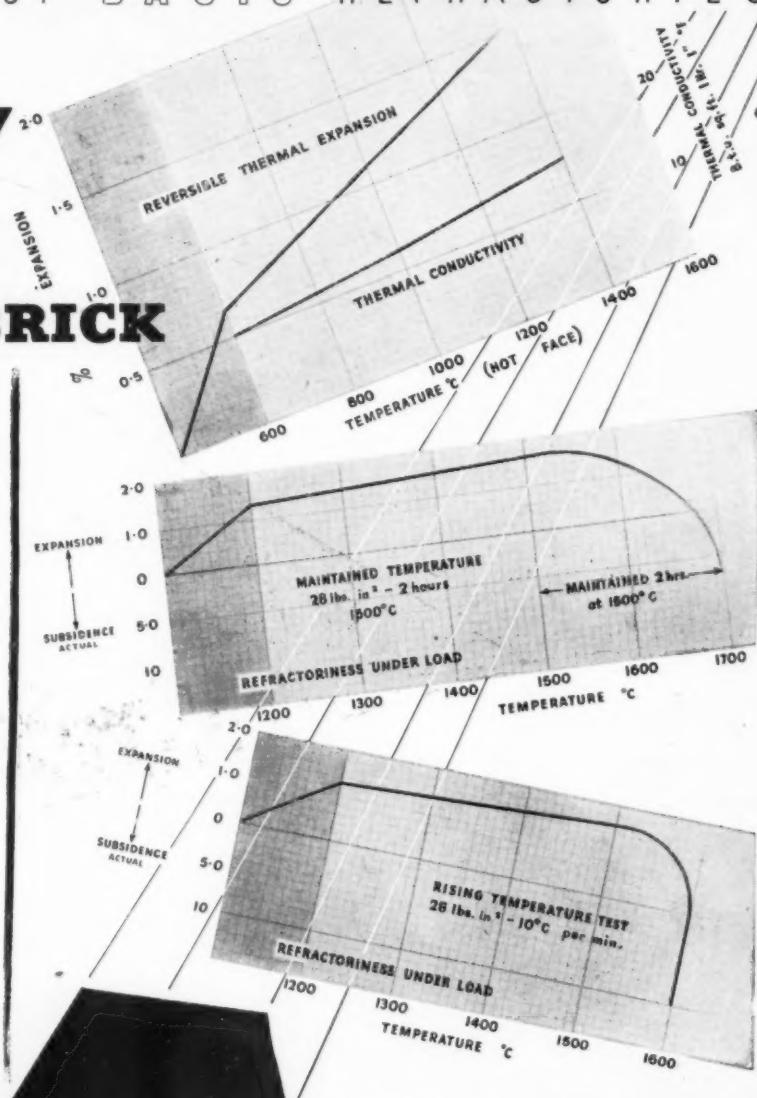
ELECTRIC FURNACES G.R. '341' dolomite bricks are used extensively in walls and bottoms of basic electric arc furnaces. Proved the most economic basic brick available. Complete absence of chromic oxide makes '341' most suited for production of chromium-free alloys. Any size furnace can be lined from standard stock sizes.



BASIC BESSEMER CONVERTERS G.R. '341' bricks, dense and burnt to vitrification at high temperatures are superior to rammed tarred dolomite or 'green' tarred blocks in the lower wall positions. In addition to giving longer and uniform life to the converter the efficiency of the process is greatly improved. Highly resistant to chemical erosion and mechanical attrition.



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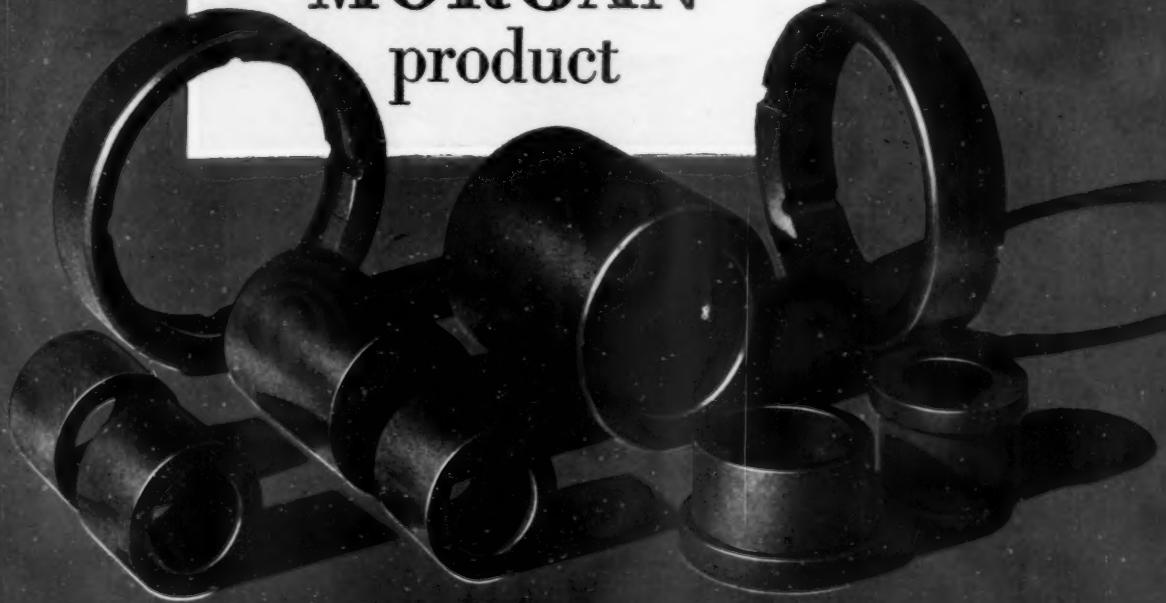
They resist conditions that are harmful to other bearings, such as lack of lubricant, the presence of chemical liquids or fumes, steam, and cyclic changes of temperature.

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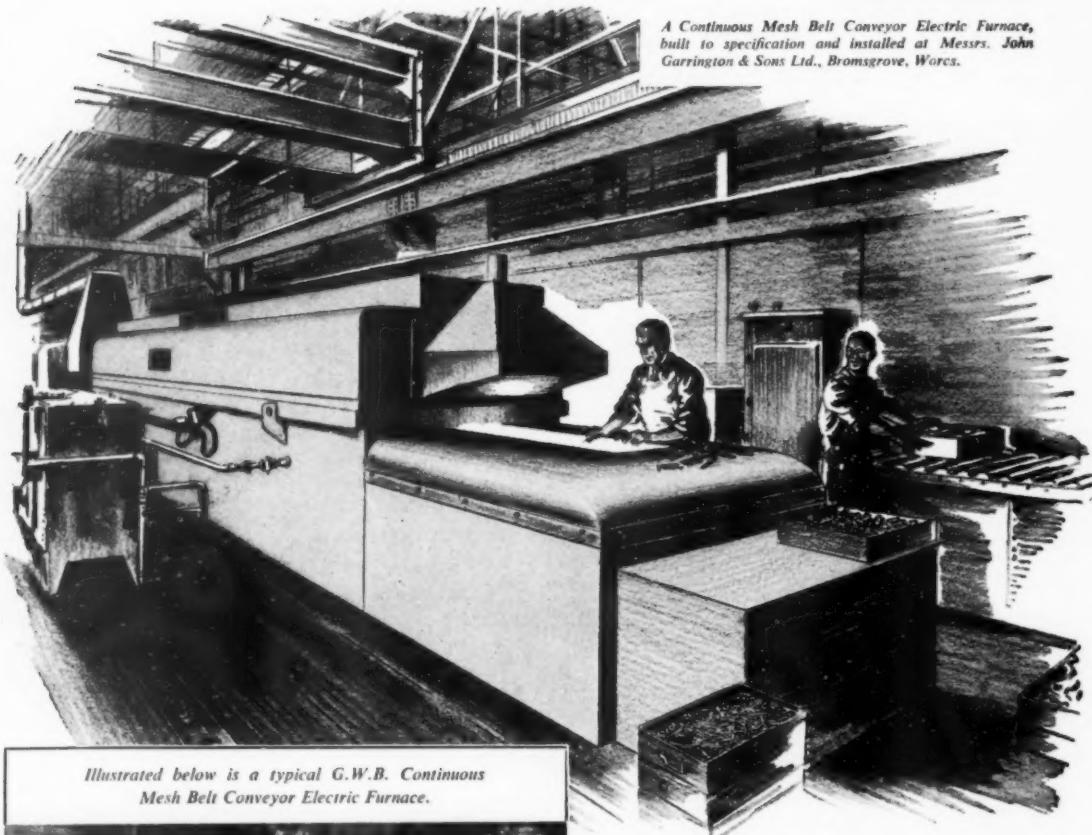
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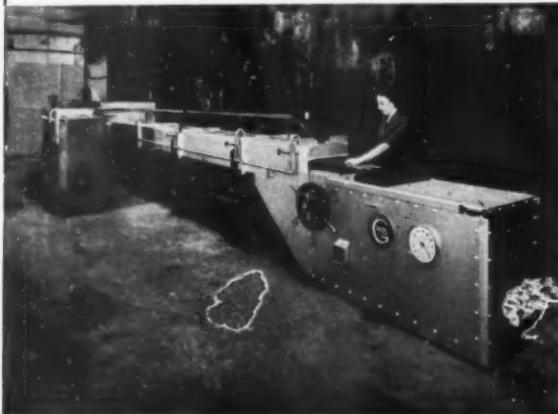
Electric Furnaces in Action

No. 2



A Continuous Mesh Belt Conveyor Electric Furnace, built to specification and installed at Messrs. John Garrington & Sons Ltd., Bromsgrove, Worcs.

Illustrated below is a typical G.W.B. Continuous Mesh Belt Conveyor Electric Furnace.



Mr. R. J. Garland of Messrs. John Garrington & Sons Ltd., says :

"The hardening and tempering of machine tools is a job that demands the utmost accuracy in control of all relevant factors, the most vital — and usually the most difficult to control — being atmosphere and materials used. This Continuous Mesh Belt Conveyor Electric Furnace seems to have overcome these difficulties to a remarkably high degree.

The furnace was built to specification by Messrs. G. W. B. Electric Furnaces Ltd., and has proved to be a sound investment. Installed in 1945, it has to date handled approximately 3,000,000 small tools of all kinds. Replacements and maintenance costs have been practically nil."



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This drawing is based on a High Duty Alloys Ltd. photograph

Nimonic is a Registered Trade Mark and the alloys sold under this mark are the subject of world-wide patents.

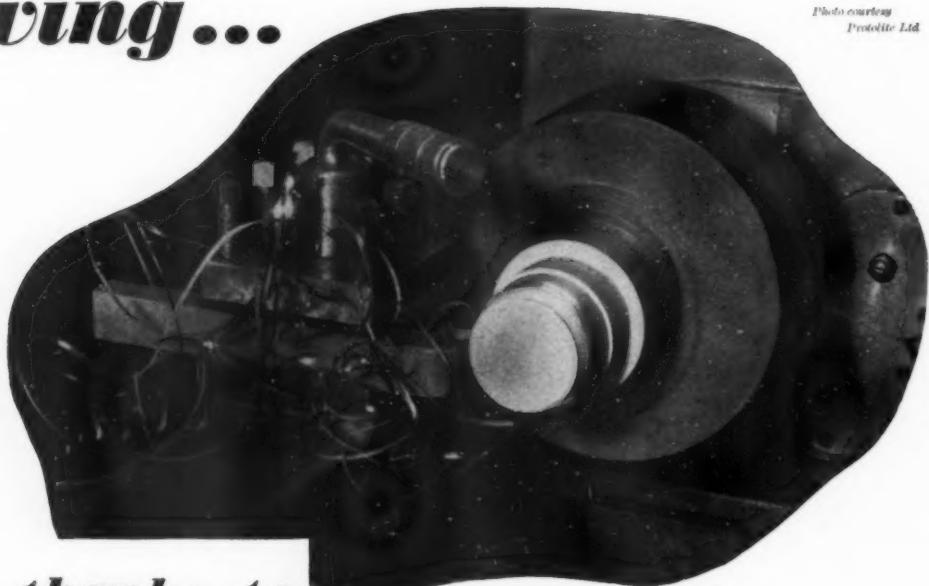
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having...*

*Photo courtesy
Protolite Ltd.*



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Here is a way in which H. F. Heating reduces cost and directly aids the production drive—brazing carbide cutting tools capable of making heavy cuts and speeding output. This is only one of the many ways in which induction and dielectric heating is helping British manufacturers beat production snags and improve quality.

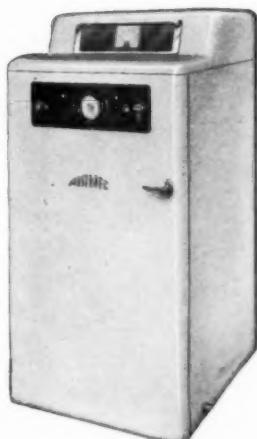
Soldering, annealing and tempering, case hardening, powder metallurgy, dehydration, sterilisation, preheating moulding materials, thermoplastic welding, adhesive setting are just a few of the ways in which an Airmec Electronic Heat Generator can be used to reduce cost. Have you thought about this method for your plant? List GA157 explains fully the high standards reached in our equipment.

We will gladly send you a copy and follow it with advice on any production problems you may care to raise. Please do not hesitate to ask.

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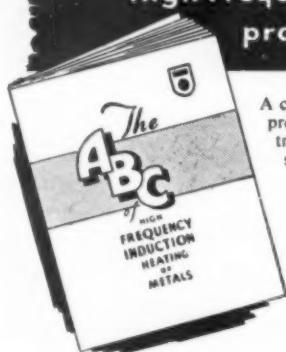
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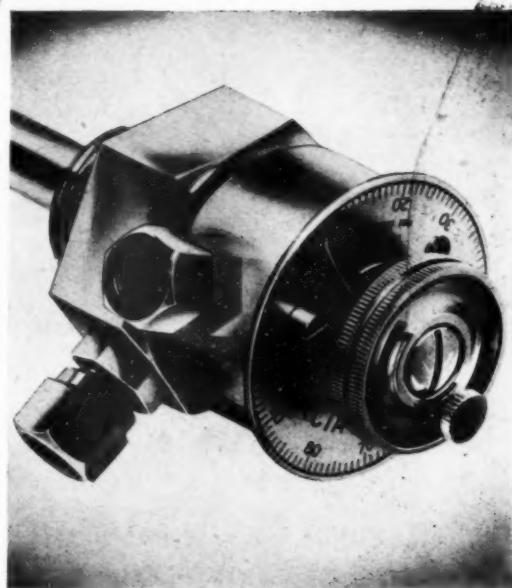
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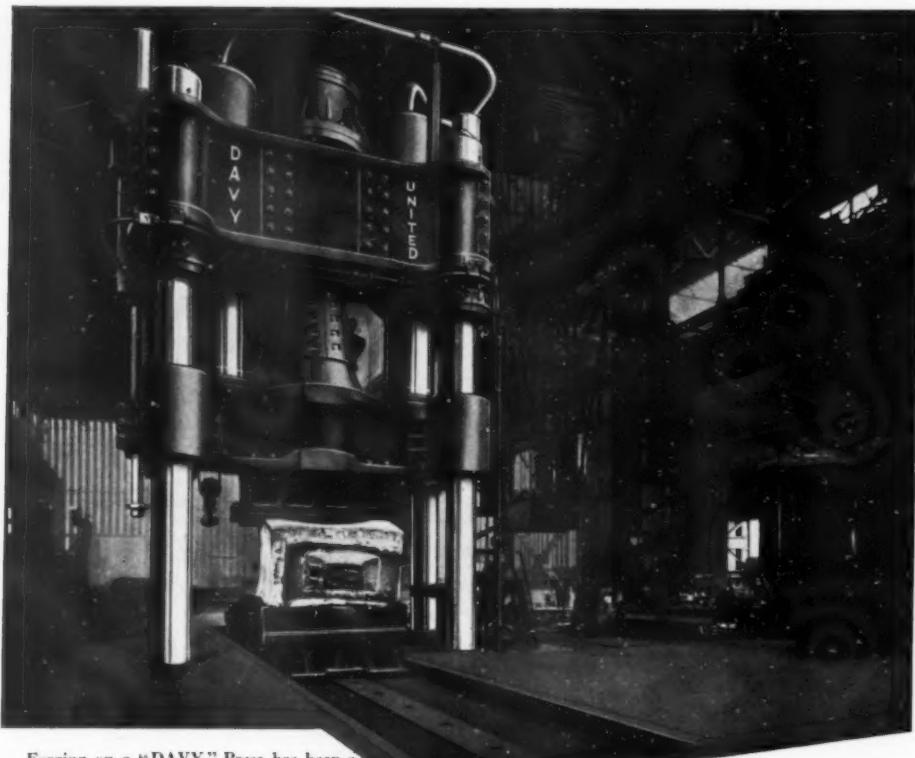
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Machinery for the deformation of metals represents the principal activity of the Davy-United Group, to-day the country's largest makers of steel and non-ferrous works plant. It was in the Glasgow Works of the Group that this press was designed and built.



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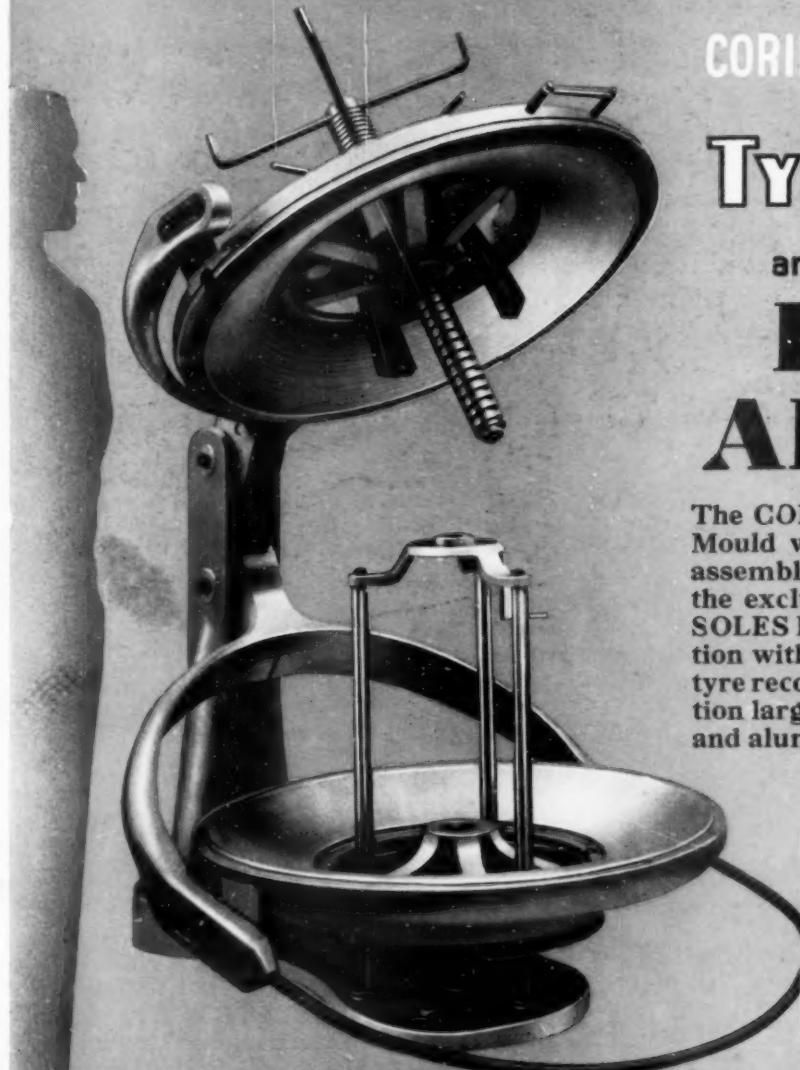
**KENT
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The CORINTHIAN Retreading Mould was cast, machined and assembled by KENT ALLOYS to the exclusive design of TYRE-SOLES LTD. for use in conjunction with their patent process of tyre reconditioning. The production largely involved aluminium and aluminium bronze castings.

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you*

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PARAMOUNT CONSIDERATIONS: Sound production, with controlled grain-flow; brought through to dimensional precision and correctly heat treated.

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VERSATILE

Suitable for welding aluminium, magnesium, copper, nickel and their alloys also stainless steel

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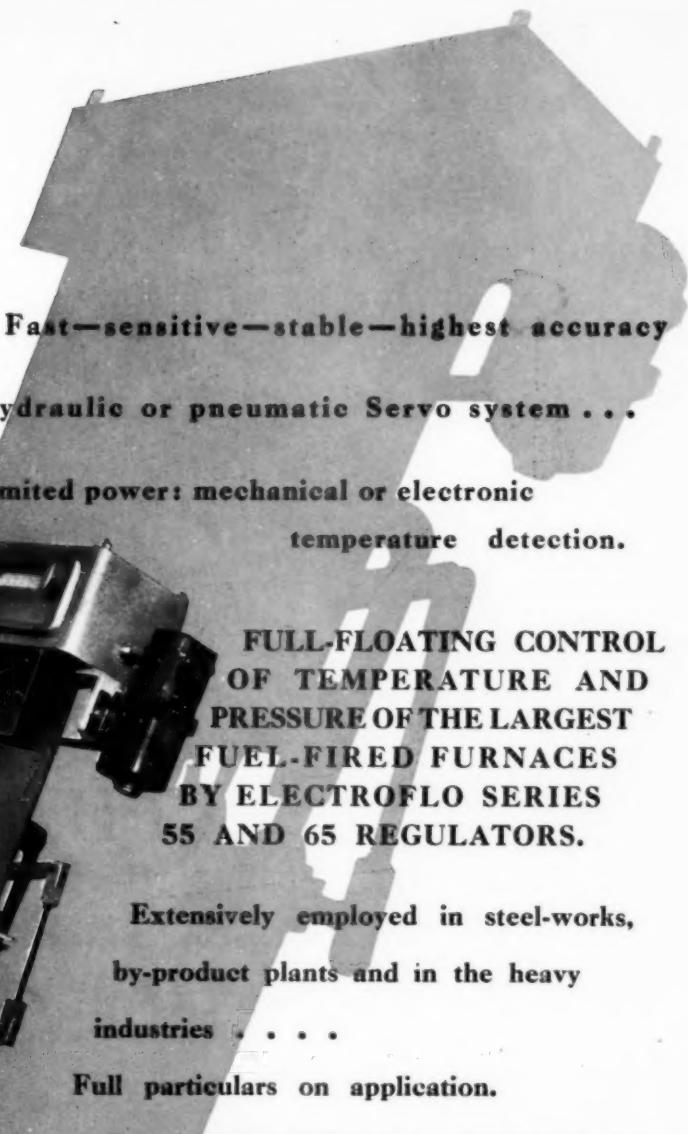
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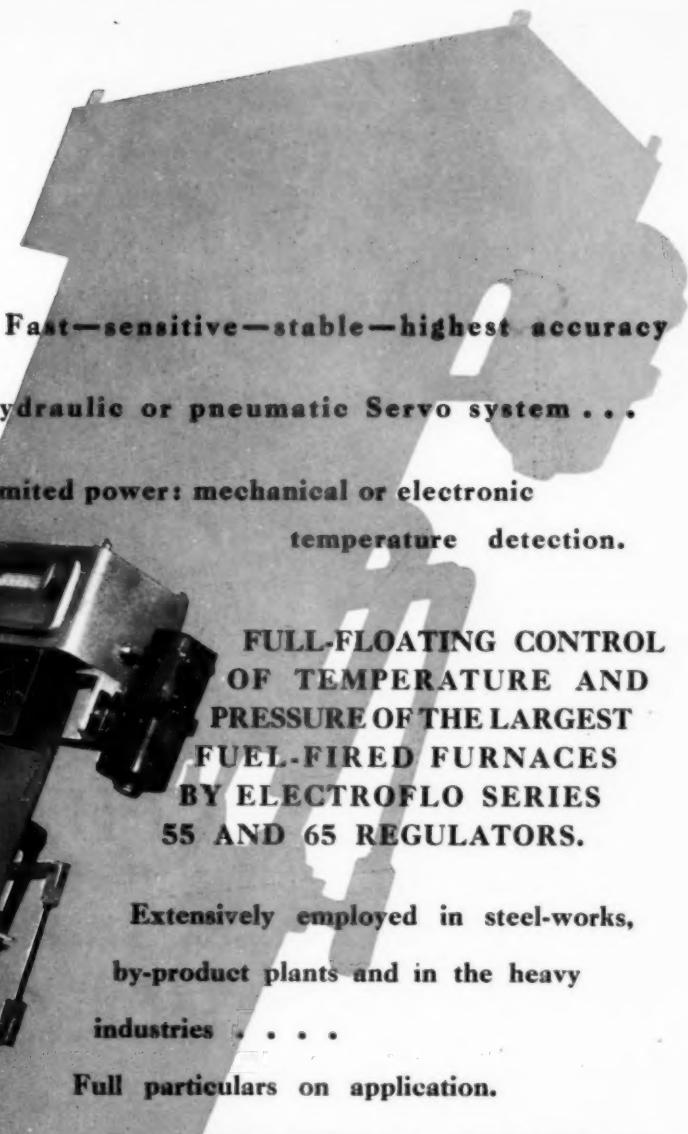
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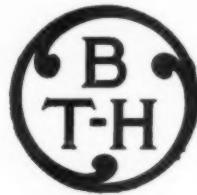
Full particulars on application.

FULL FLOATING CONTROL
by
ELECTROFLO
METERS COMPANY LIMITED

Advt. of: ELECTROFLO METERS COMPANY LIMITED, ABBEY ROAD, PARK ROYAL, N.W.10



An aerial view of the main works at Rugby.



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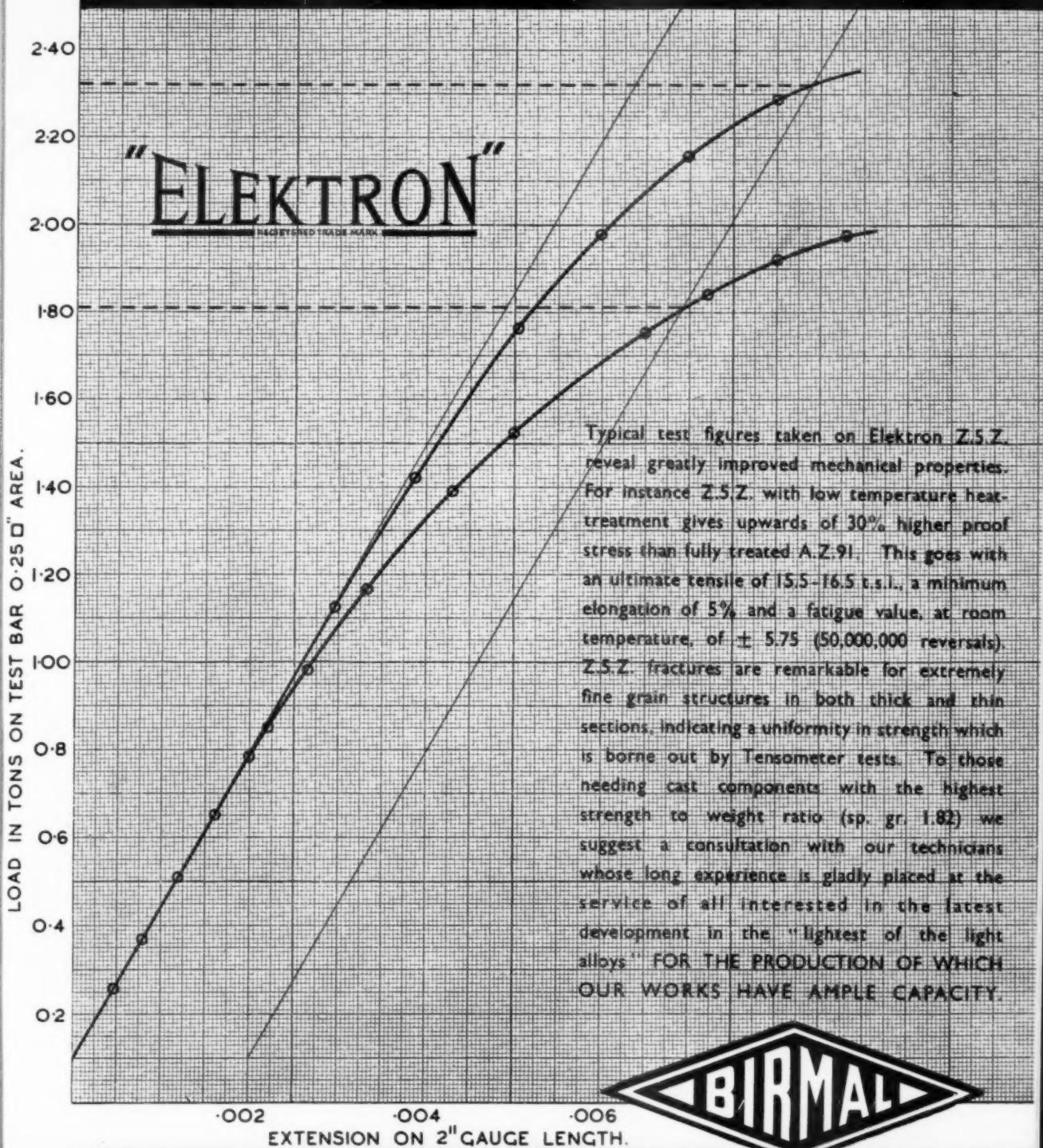


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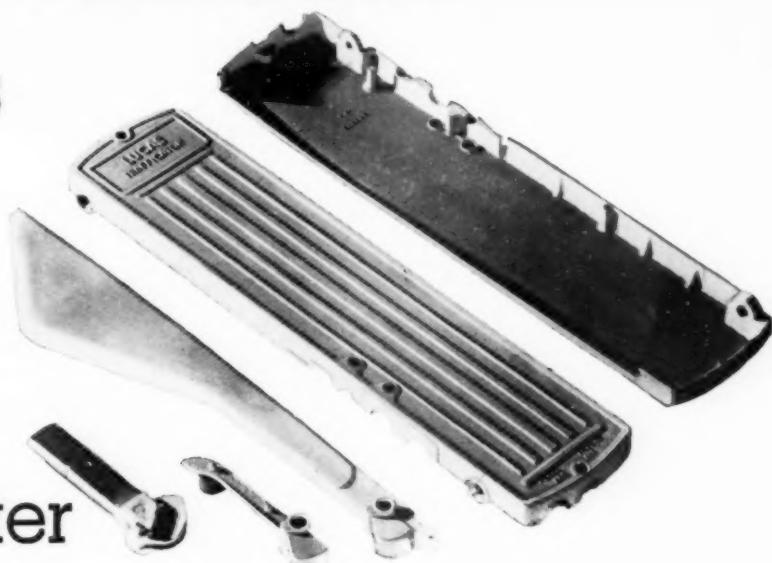
Whale catchers under construction. Photo by courtesy of Smith's Dock Co. Ltd., Middlesbrough.



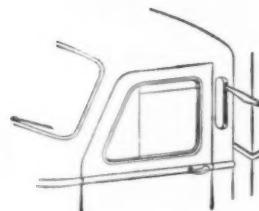
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Perhaps we're biased
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parts
are greater
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this whole



THE MODERN knights-errant who thunder down the Great North Road put their faith in the perfect working of these zinc alloy die castings, for they are components of the traffic indicator of a lorry.*

Why the parts are zinc alloy die cast

On the right are the two halves of traffic indicator casing : notice the overlapping lugs to ensure accurate mating, and the thin section strengthened by ribs. The die cast insert in the plastic arm is an interesting example of the combination of castings and plastics. The casting acts as fulcrum and stiffener, and the checkering helps to provide a key between metal and plastic. When the casting has been moulded into the arm, the cored pivot hole is made accurate to within 0.0005 in. The die cast headpiece cast onto the mild steel plunger is a fine example of a zinc alloy attachment.

* Photograph reproduced by courtesy of Joseph Lucas Ltd.

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Alloys conforming to B.S. 1004 should be specified for all applications where strength, accuracy and stability are essential.

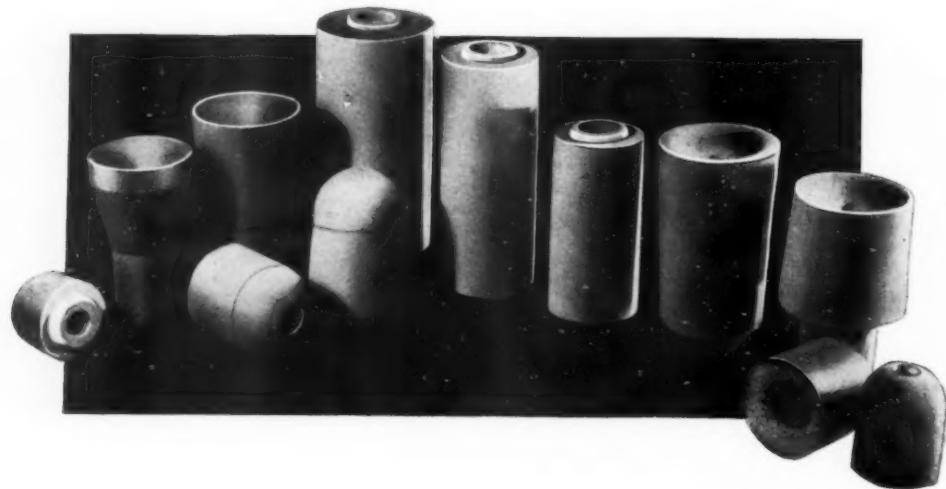
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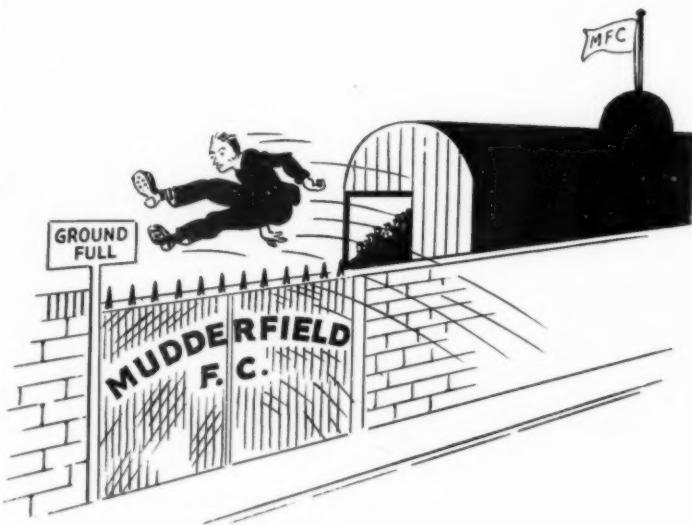
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Number Ten

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And he certainly proves with this flying invasion
How a minimum weight can rise to the occasion.



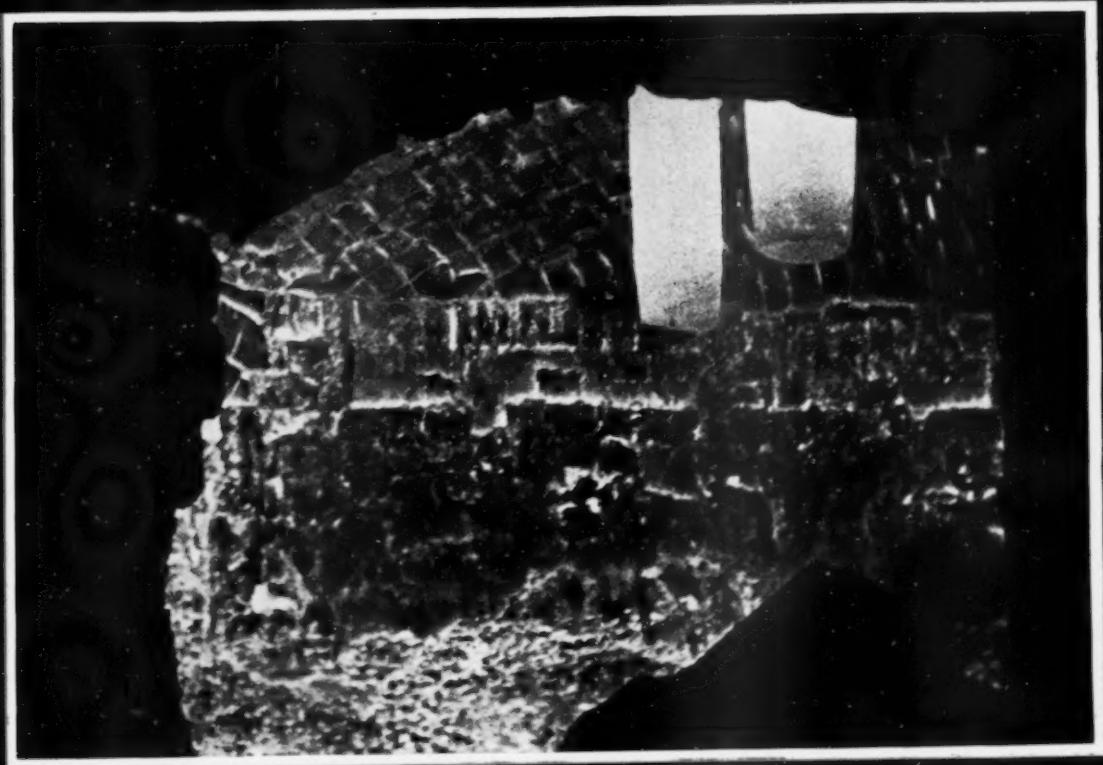
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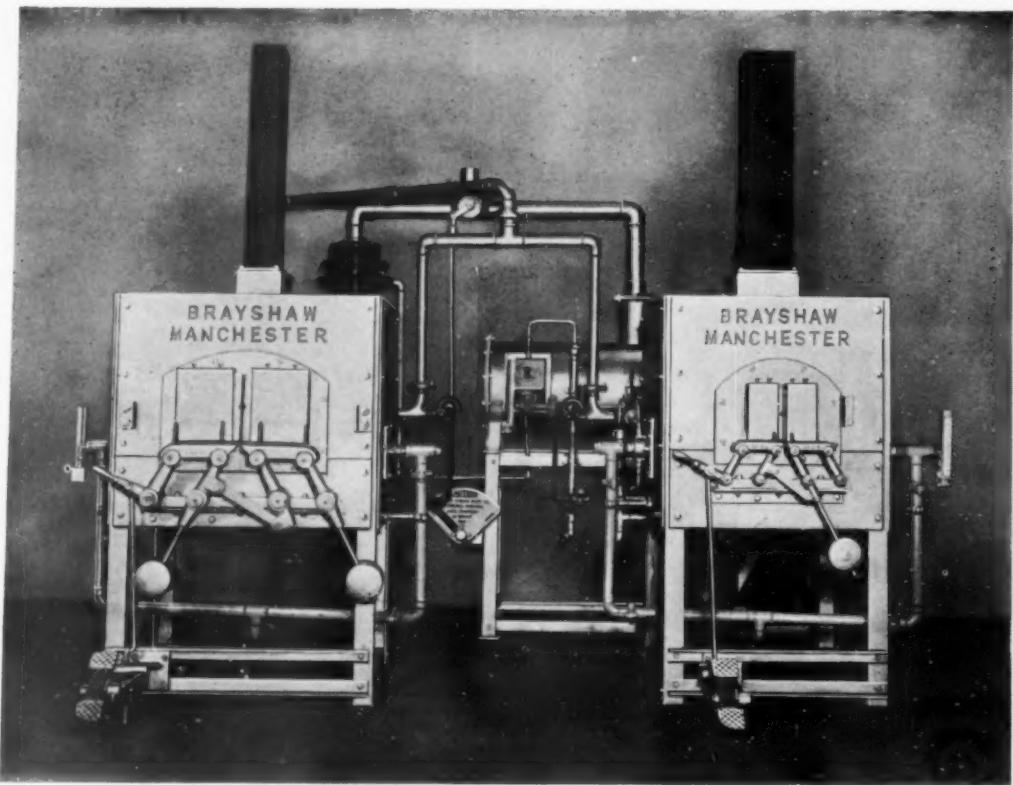


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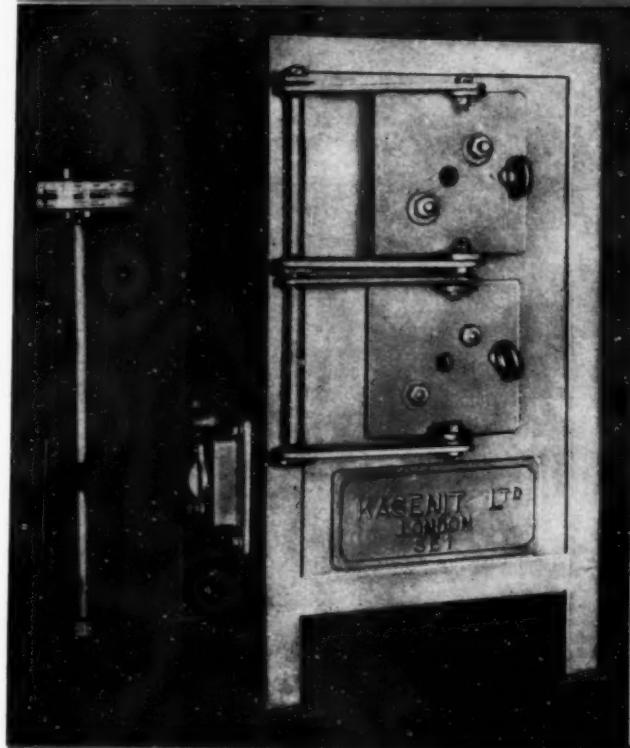
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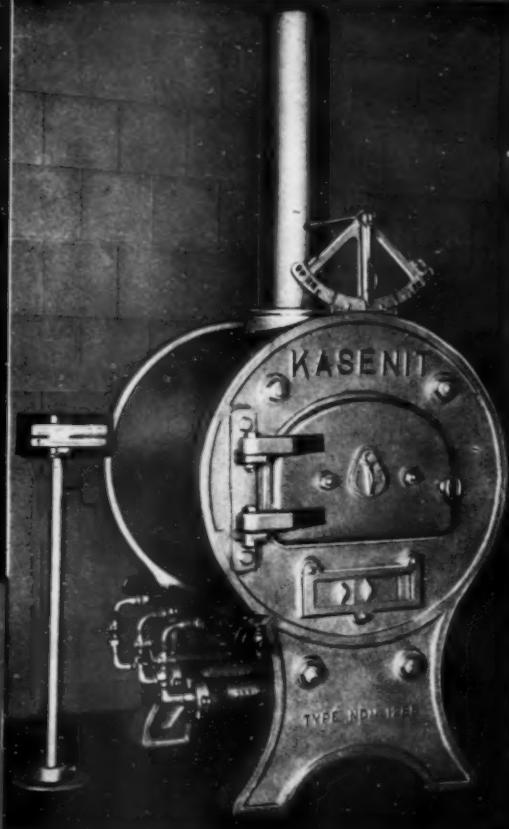
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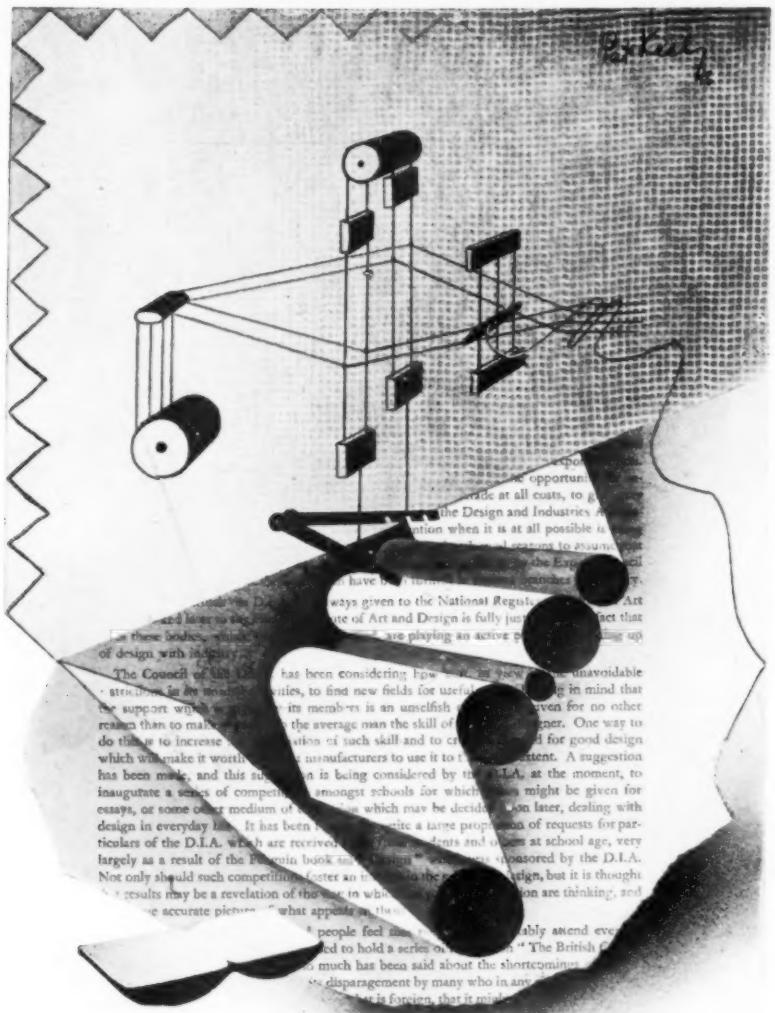


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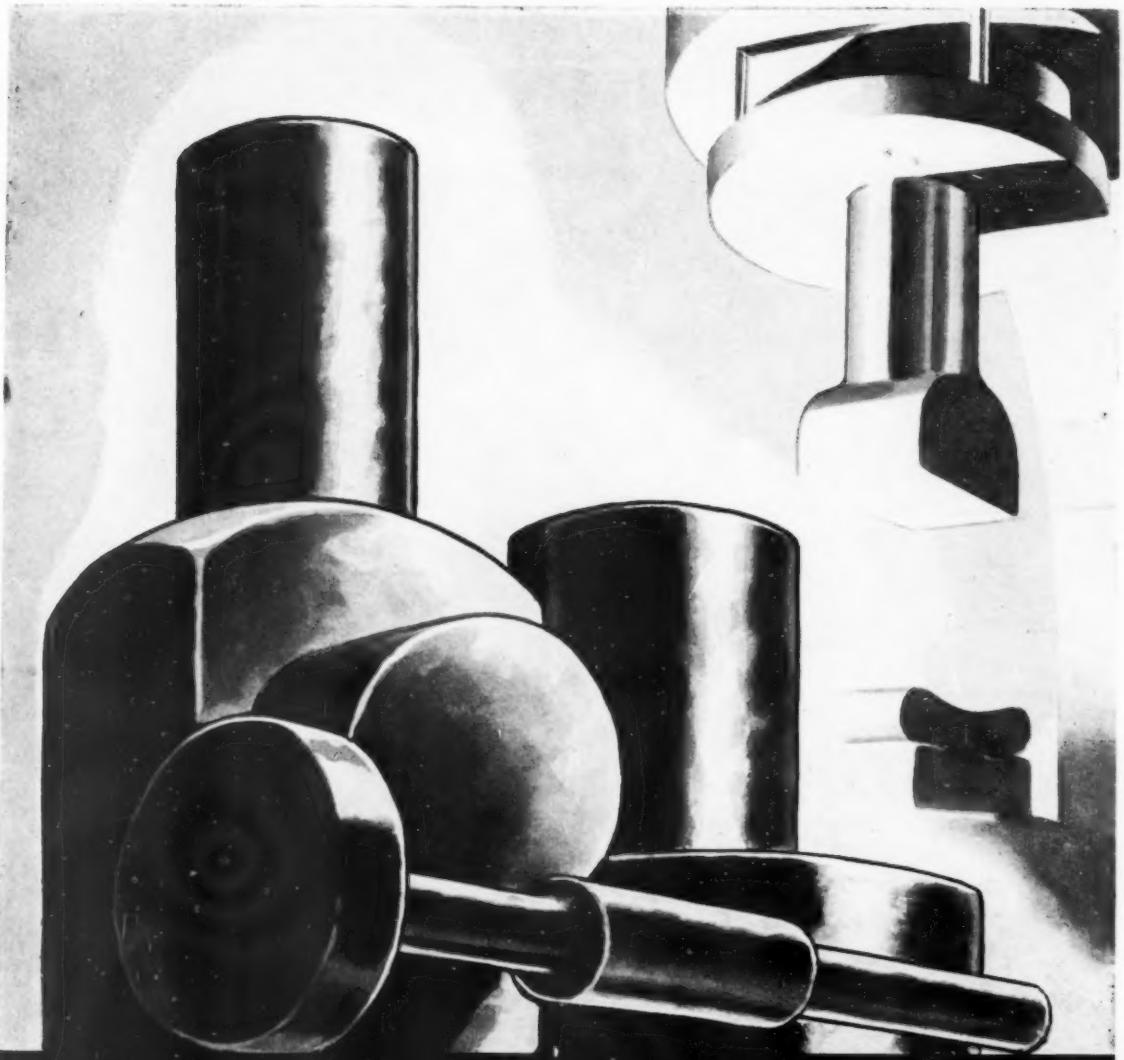


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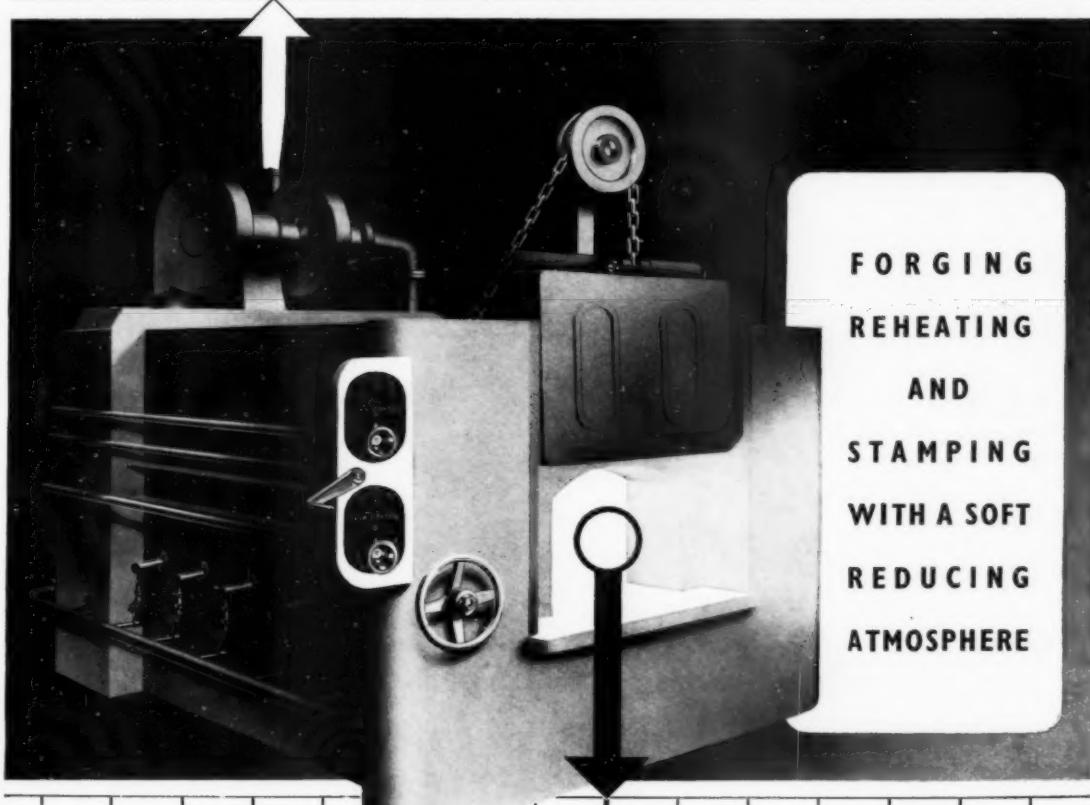
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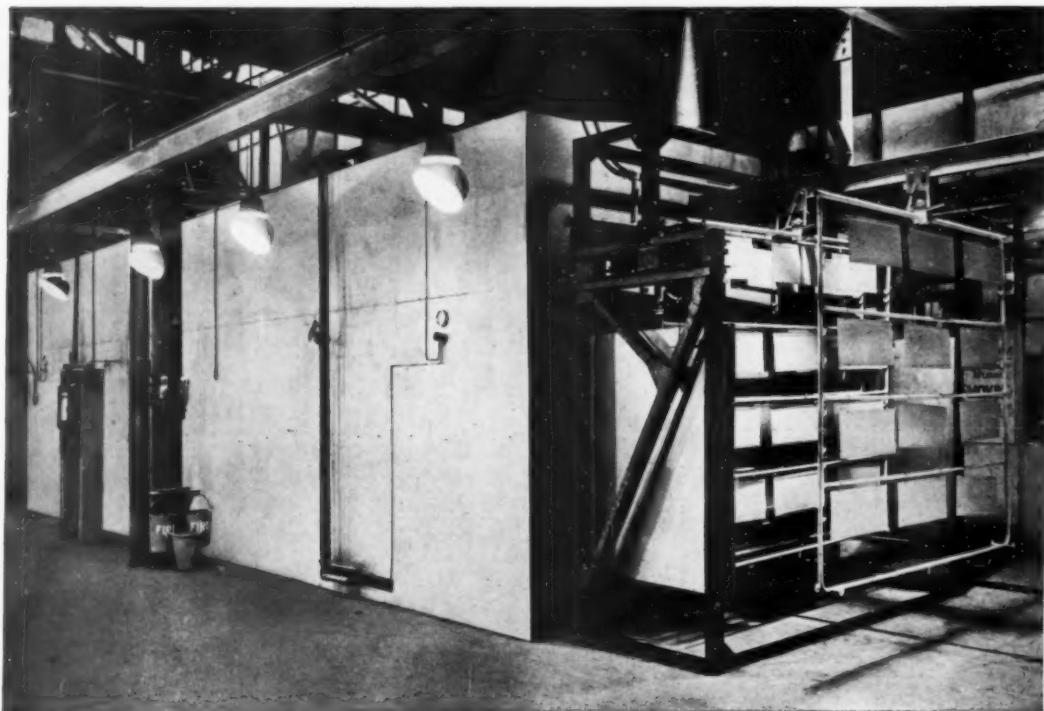
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THE BRITISH JOURNAL OF METALS.
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APRIL 1949

Vol. XXXIX No. 234

Increased Productivity

THE Economic Survey for 1949 and various aspects of the recent Budget stress what has been stressed since the end of the war, that greater emphasis must be laid upon increasing productivity, but it is not easy to see how this can be done. It is true that remarkable progress was made last year, and a very significant change made in the balance of payments. The deficit of £630 million between our foreign earnings and our foreign expenditure at the end of 1947, practically all of which resulted from trade with the North and South American countries, was very substantially reduced. During 1948 this deficit was brought down to £120 million; indeed, during the second half of the year there was a surplus of about £30 million. But we have not yet solved the problem of paying the American countries and our man-power and resources have not yet been developed to meet the changed conditions of the post-war world.

Everyone who has studied Britain's national economy realises the necessity of increasing home production of dollar-saving foods and materials; more exports to the American countries; the diversion of purchases from hard-currency to soft-currency sources; the encouragement of dollar-earning activities in the Commonwealth and Colonies; and the increase of exports to sterling area countries likely to help in the saving of dollar expenditure. Some progress has already been made along these lines, and as a result the pattern of imports has approached nearer to that of pre-war.

On the question of exports, the position is not nearly so good. Our exports to the American countries have increased, but by less than the average of all exports. Thus, while exports are increasing, their distribution is anything but satisfactory. That is probably why the Survey emphasises the need for increased exports to the United States and Canada as the first of the objectives for 1949. But the achievement of this objective depends largely upon increased production and upon cheaper production, which are objectives two and three. These markets are available for the right products for sale at the right prices. Britain, of course, is not the only country seeking a balance of payments by increasing exports and with European recovery progressing, competition in our export markets is becoming much keener, in terms of prices, designs and deliveries.

One of Britain's greatest problems is man-power. Her great schemes to improve general educational facilities, raise the standard of living, help the sick and aged, reduce working hours and bring about better working conditions, at a time of unstable national economy, has become a heavy burden for industry, and, with the numerous controls, has absorbed a great amount of unproductive man-power, all of which must be supported by earnings. Indeed, it is extremely doubtful whether any other industrialised country carries such a high

proportion of unproductive man-power. Yet we need more coal, higher agricultural output, more textiles, and more steel. None of the 1948 man-power targets for these industries was even approached. Thus, we are still a long way from what can be regarded as a balanced industry to satisfy our economy and the going must increase in severity with time, unless man-power can be increased or output raised from present man-power.

It will have been noted that the export targets for 1949 are more conservative than those set for 1948. It is an acknowledgment that the markets have become more critical of prices, designs and deliveries. Targets for the end of this year are only 155% of the 1938 volume, although this percentage was nearly achieved during the last quarter of 1948. But it should not be assumed that there can be any relaxation of effort to increase exports.

The suggested targets assume that world markets will continue to need products that we can make and that they will be in a position to take them. Of course, as far as internal needs are concerned there is an immense demand for our own products, which could be readily created by reduced taxation, and there is a tremendous amount of work to be done, but, since the majority of our food and raw materials must be imported, there is no likelihood of Britain ever becoming self-sufficient, even if she desired to be. Payment for these imports can only be made by exports. Obviously, any depression in the rest of the world would result in a fall in exports and throw us back on our own resources. This would be a calamity and the warning of Mr. E. H. Lever, Chairman of Richard Thomas & Baldwins, Ltd., in a speech to representatives and executives of the Company, is to the point. He said, "growing demand will only become a reality if the world recovers its sanity and realises that it is only by hard work and creative effort by us all and by the creation of conditions which will induce men and women to give of their best that we can avoid one of the greatest economic collapses the world has ever seen. Such a collapse, if it came, might result in such a social and economic upheaval that generations might pass before a new stability could be created." We have practically nothing to cushion the effects of such a calamity. Our savings invested abroad were largely consumed to finance the recent war and to-day we are in the position of living from hand to mouth. Only a prosperous world, able to buy our products, will permit us to restore our finances so that fluctuations in world trade would not have such serious effects and hard work, by all, would be a great help.

The Survey is wholly concerned with the problem of restraining inflation and it has helped us to understand the background against which the Budget has been worked out. This year is apparently intended to be a year of consolidation and increased productivity is an important essential to this end and must continue to be emphasised.

Improvements in Precision Balances

THERE are few industrial processes into which the measurement of weight does not enter at some time, and variations of the standard spring or beam balances are many; the differences in capacity are also considerable and the sensitivity varies with capacity, the most accurate being those installed in laboratories in connection with which a recent display by L. Oertling Ltd. proved of great interest.

Over 100 years ago, L. Oertling commenced in London as a maker of analytical and assay balances. Continued advances in design have enabled the firm to place British precision balances in the forefront of the world's finest; they have led to to-day's sensitive, quick reading and in many cases semi-automatic models, including improved beam design and rider action, the introduction of clamping devices, of direct scale reading and of automatic weight control. It is remarkable, however, that over a period of a century surprisingly little development has taken place in the design of their all-important bearings. Tests carried out in this company's research laboratories have shown that occasional and apparently inexplicable "sticking" and irritating small inconsistencies in rest point readings may be due to minute variations in the physical properties of the agate planes, resulting in a departure from optical flatness after prolonged service. A direct development from these tests is the adoption of Corundum as a new material for the planes—a material much harder and more uniform than agate.

Corundum is essentially crystalline alumina which can now be prepared in large single crystals of uniform hardness, elasticity and brittleness. The hardness, second only to diamond in the mineral field, is 9 on Moh's scale as compared with 7 for agate; moreover, the latter varies from point to point on the plane with the chemical composition and structure of the natural stone, whereas the single synthetic crystals from which the new planes are cut are uniform throughout.

Success in the preparation of the Corundum surface in the optically flat condition has demanded many months of painstaking research, but the finish now being obtained completely satisfies the stringent optical tests which all Oertling balance planes have to pass. Carefully conducted endurance tests have shown in ample measure the improved performance secured by the extra hardness and uniformity of Corundum planes. It is considered to be the most important single advance for many years and Oertlings are now fitting these Corundum planes as standard on their complete range of prismatic, analytical balances at no extra cost.

Many readers will be familiar with the Oertling prismatic-reflector device whereby an enlarged image of a graticule fixed to the end of the pointer is projected on to a ground glass screen. Work has been carried out with the object of improving the brilliance and precision of the reading index and now a completely redesigned optical system is being fitted to all prismatic balances. The result is a very brilliant, well-defined image easily read in full daylight and from any position in front of the balance case.

Although the foregoing make the main technical advances displayed, other exhibits were not without interest, particularly the method of heat-stabilising, applied to all Oertling balances being shipped to or through the tropics, nickel-chromium weights, and an electrolytic method applied to the testing of rhodium-

plated weights. The general range of standard models on view, comprising chemical, semi-micro-chemical and microchemical balances, is noteworthy for its general simplicity and cleanliness of design.

Industrial Finishing

DESPITE the attention directed to the importance of finish, during recent years, and to the development of many processes it is not yet fully recognised that, where competition is encountered in the sale of metal articles or articles carrying metal parts, the quality and attractiveness of the finish represent selling points, the importance of which cannot be over-emphasised in any market. Many manufacturers still fail to realise that finish constitutes by far the initial sales appeal. It is, after all, the appearance of the article which first catches the eye of a potential buyer whether at home or abroad, and the way in which the finish wears largely determines the customer's satisfaction with his purchase and the extent to which repeat orders will result. In some instances superior finish may be used to cover unsuitable material or inferior workmanship but failure must eventually result from such practice.

It is true that many manufacturers appreciate the importance of finish and regard it as an integral part of production, but much progress has yet to be made before the general standard is raised to the level likely to lead to the success sought in what is now a buyers' market. There is no lack of technical ability to achieve this result and excellent finishing processes are available, but technical information on the subject is not fully used by all those trades likely to profit by it; yet at no time has metal finishing assumed so much importance as it does to-day, especially with products for overseas markets.

At a recent conference, where representatives of commercial organisations, trade associations, and research societies met, there was unanimous agreement that bad finish, to a large extent, was due to ignorance and not to bad workmanship. Fortunately, more direct attention is now being focussed on this question of finish and the Industrial Finishes Exhibition, to be held at Earls Court, London, from August 31st to September 13th, should do much to show the wide range of finishes available, and the means by which they can be applied. The displays at this exhibition will enable users of finishes, the distributive trades and the general public to see, for the first time under one roof, the great variety of finishes, processes and their comparative qualities, as well as many new developments which are not widely known.

Every branch of the finishing trade will be represented at this Exhibition, including chemical finishes, vitreous enamels, organic finishes, electro-deposition on precious and base metals, and natural finishes on metal surfaces. Thus, there will be non-metallic coatings such as anodising, phosphating, etc.; vitreous enamels on iron, steel, alloys, etc.; and paints, enamels, varnishes, lacquers, resins, rubber, etc.; polished, matt, machined and hammered natural finishes; platinum, gold, silver, palladium, indium, and rhodium platings; base metal platings in chromium, cadmium, copper, lead, nickel, zinc, and various alloys; dipped and sprayed metal coatings in tin, zinc, lead, nickel, etc. Attention will also be directed to preliminary cleaning, pickling and polishing operations, and to plant and equipment.

This exhibition should go a long way to increase the production of really first quality products.

The Relaxation Test in Terms of Creep and Creep Recovery

By E. A. Johnson, M.Sc., M.Sc.Tech., A.M.I.Mech.E.

(Communication from the National Physical Laboratory.)

Previous work has shown that in the two cases of a low carbon steel and a chrome molybdenum steel the behaviour in a relaxation test cannot be directly ascertained from data obtained in normal creep tests. This fact may be due to the combined effects of several factors, among which is creep recovery (which in the relaxation test is necessarily associated with the gradual reduction of stress). The current paper describes an effort to ascertain the relative importance of creep recovery as a link between the relaxation test, and normal creep tests. Relaxation tests from relatively high and relatively low initial total strains have been made on a 0.17% C. steel at 445° C., and on a chrome molybdenum steel at 485° C. Estimates of creep recovery occurring during the relaxation test were made by means of completely unloading the specimen at intervals during the test. Adjustment of the values of measured creep rates in the relaxation test was made to make allowance for the effects of the negative recovery strain and the modified relaxation creep rates compared with rates computed from auxiliary creep tests by means of an assumption of the time hardening and strain hardening theories of plastic strain.

Two papers^{1,2} recently prepared for the J/E Committee of B.E.I.R.A., the present author showed that the behaviour in the relaxation test* of a 0.17% C. steel, at temperatures from 425°–485° C., and of a chrome molybdenum steel at temperatures from 455°–525° C. could not be estimated from normal creep tests on the basis of either the time hardening or the strain hardening theories of creep.

Correlation of creep and relaxation tests requires a knowledge of a number of factors which control the plastic properties of the material in question. If complete knowledge of these factors were available, it should be possible to use the results of one type of test to predict the results of a test of different type and possibly to obtain the same information from one relaxation test as from a group of creep tests. Among these factors it is obvious that strain hardening, temperature softening, creep recovery and possibly others play their part. The usual time and strain hardening theories of creep behaviour are simplifications not directly applicable even in creep tests involving step down loading and certainly do not apply directly in relaxation tests as shown by the author (see above). It appeared reasonable to examine the effects of the element of creep recovery which is associated with the relaxation test in view of the continuous reduction of stress occurring in this test.

* A relaxation test is here defined as a test in which the total strain incurred upon applying the initial full load, is subsequently maintained constant by reduction of the current load.

It is obvious that for every decrement of stress in the relaxation test due to creep strain, some element of creep recovery must in general occur. This recovery or negative creep strain will tend to reduce the total creep strain occurring in the relaxation test, so that under relaxation conditions the creep rate at a particular relaxed stress might always be expected on these grounds to be smaller than that corresponding to that stress under normal conditions of creep. The numerical estimation of the effect of the recovery strain is however, a matter of very considerable difficulty. It has been shown in a previous paper† by the present author that for a 0.17% C. steel the creep recovery $R(t)$ at time t after unloading in a normal creep test is

$y_0 [n \log (t+1)]$ where y_0 is the initial elastic strain on unloading, n is a constant depending upon temperature and period of previous creep, and t is the recovery period in hours.

The above expression may be rendered

$$R(t) = \frac{(S_0 - S)}{E} [n \log (t+1)]$$

where S_0 and S are the initial and final values of the stress, and for an elemental stress decrease

$$R(t) = \frac{\delta S}{E} [n \log (t+1)]$$

Now under relaxation conditions the reduction of stress is not a single action as in unloading a creep test, but proceeds gradually so that for any

current time t_0 , the preceding creep strain has not been under steady stress, but under diminishing stress. Nevertheless some indication of how the recovery strain affects the relaxation may be obtained by summing the effects of such elements of recovery as

$$\frac{\delta S}{E} [n \log (t+1)]$$

from the beginning of the relaxation test until the current time. This leads to a complex addition to the right hand side of the equation

$$\text{of relaxation } x = \frac{S - S_0}{E} \text{ (where } x \text{ is the}$$

creep strain in the relaxation test) and the subsequent development of a time-relaxed stress equation involves very considerable mathematical difficulties and one is forced to the conclusion that a strictly analytical approach to the effect of recovery is not easily possible. This being so, the most reasonable approach to the problem of estimating the effects of creep recovery during the relaxation test must be by means of a direct or indirect measurement of the recovery occurring during the relaxation tests. This is the method which has been adopted in the currently described tests.

In the present work, relaxation tests have been made on a 0.17% C. steel at 455° C., and from initial total strains of 0.00588 and 0.00054, and on a chrome molybdenum bolt steel at 485° C. from initial total strains of 0.00172 and 0.0005. The chosen initial total strains were relatively small and large in each instance. These tests were continued for 10,000

† Proc. Inst. Mech. Eng., 1940–41, 145, 210–212.

minutes, being interrupted at suitable intervals to unload completely and measure the creep recovery corresponding to the complete unloading decrement, from which could be computed the recovery corresponding, at the current period of test, to the decrement of stress relaxation in the relaxation test*. The period of recovery in each case was approximately equal to that of the relaxation test, viz., 10,000 minutes less the period of unloading. The test temperature for each material was chosen as being one at which no appreciable softening of the material occurred over long periods. After each recovery period, the test was allowed to creep under the current load until the total strain was equal to that initially imposed, this total strain was then maintained constant by relaxing the load further.

The modification of creep rates measured in the relaxation tests to take account of the effects of recovery was then estimated, and compared with creep rates based on the assumption of the theories of time hardening and strain hardening and by use of auxiliary tensile creep test results.

To provide data for interpretation of relaxation tests in terms of the time hardening and strain hardening theories, a series of creep tests at various stresses covering the range concerned in the relaxation tests was made for the two materials at the temperatures concerned. These tests again lasted 10,000 minutes.

Particulars of Materials

The 0.17% C. steel used was in the form of hot rolled bars $1\frac{1}{2}$ in. in diameter. It had been produced in a basic open hearth furnace, its percentage chemical composition being as follows: carbon 0.17, manganese 0.72, silicon 0.22, sulphur 0.024 and phosphorus 0.022. Before machining test specimens from the material, suitable lengths of the bars were heated for $\frac{1}{2}$ hour at 950°C , and then air cooled. The material was in general, similar to that used in the tests of Ref. 2, but the bar size being somewhat different, the creep characteristics varied somewhat from those found in the auxiliary creep tests of Ref. 2 and it was necessary to conduct a further set of auxiliary tests to interpret the current relaxation tests.

The chrome molybdenum steel was in the form of bars $1\frac{1}{2}$ in. in diameter. The percentage chemical composition was as follows: carbon 0.42, silicon

0.19, manganese 0.61, sulphur 0.036, phosphorus 0.022, chromium 1.41, nickel 0.11, and molybdenum 0.78. Before use, the material was hardened in oil from 850°C , followed by tempering at 640°C .

Experimental Procedure

The relaxation tests were made in high sensitivity creep testing units of 5 tons capacity, loading being by means of a double lever system and jockey weight. The test pieces used were 0.564 in. diameter, and of effective gauge length 5.3 in. Test temperatures were maintained constant to $\pm 1^{\circ}\text{C}$ and the temperature gradient along the gauge length was reduced to 3°C .

The initial strain was measured and the total strain during test maintained constant by use of the normal high temperature creep extensometer, the loading being controlled by hand. This was made possible by the fact that interruptions of the test for measurement of recovery (referred to in detail below) were so spaced that there was no need for automatic control during periods of actual relaxation.

At more or less equal decrements of relaxed stress the load was removed entirely and the recovery observed over 10,000 minutes*. This period (equal to that of the whole relaxation test) was chosen in view of the fact that for each decrement of relaxed stress it was desired to find the effect of recovery over the remaining portion of the relaxation test, the sum of such effects being regarded as the total effect of recovery. The recovery due to the actual relaxed decrement of stress of course, was equal to the quantity.

Measured recovery for completely removed load $\times \frac{\text{Relaxed stress decrement}}{\text{Total load removed}}$

The auxiliary tensile creep tests were made in creep units of 2 tons capacity using an extensometer capable of measuring extension of order 10^{-5} inches and with a furnace temperature controlled to $\pm 1^{\circ}\text{C}$.

Results of Tests

The results of the relaxation tests *in toto* are shown in Figs. 1 and 2 and in Tables V and VI, and the results of the auxiliary creep tests in Figs. 3 and 4.

In Tables I-IV, the experimentally measured increments of creep recovery due to the several decrements of relaxed stress are given. From the

* Since recovery in a given time is linearly proportional to the stress decrement.

† Less a period equal to current time of relaxation.

RELAXATION TESTS ON 0.17% C STEEL AT 455°C .

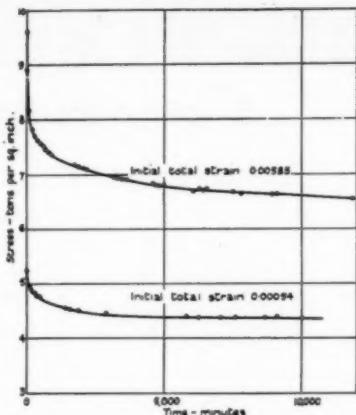


Fig. 1.—Relaxed stress - time curves from initial total strains of 0.0058 and 0.0054.

RELAXATION TESTS ON CHROME MOLYBDENUM STEEL AT 485°C .

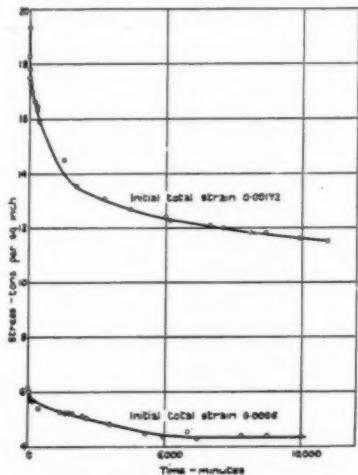


Fig. 2.—Relaxed stress - time curves from initial total strains of 0.00172 and 0.0005.

figures given in these tables the total creep recovery† for various periods of the relaxation test was computed in the manner indicated in Fig. 5. In this and similar figures (not shown) the recovery strains corresponding to each of the various periods at which the relaxation test was unloaded are superimposed one upon the other at their correct intervals of time in the relaxation test. That is to say the initial recovery curve corresponding to the first period of unloading in the relaxation test is drawn with the zero of strain set at the particular period of unloading and of course, covers the period from that unloading period to

† Including the initial unloading elastic strain.

AUXILIARY CREEP TESTS ON 0.17% C STEEL AT 455° C.

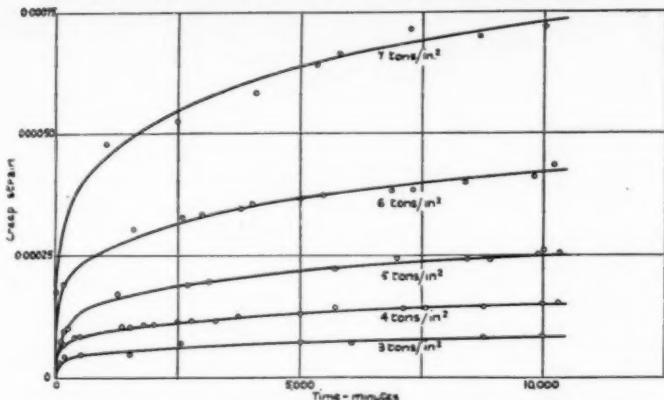


Fig. 3.—Creep strain - time curves. All creep curves conform to geometrical form of composite curve.

AUXILIARY CREEP TESTS ON CHROME MOLYBDENUM STEEL AT 485° C.

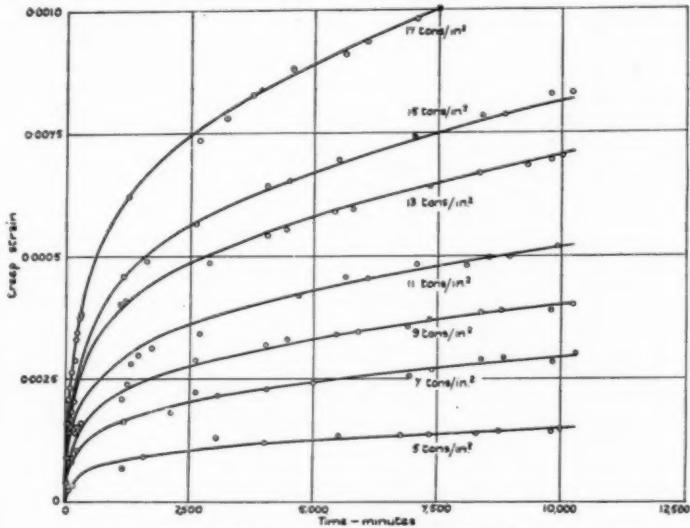


Fig. 4.—Creep strain - time curves. All creep curves conform to geometrical form of composite curves.

RELAXATION TEST ON CHROME MOLYBDENUM STEEL AT 485° C.

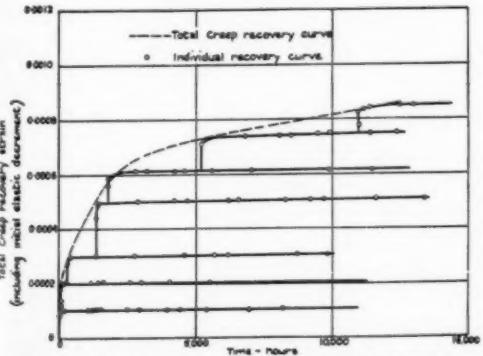


Fig. 5.—Total creep recovery - period of relaxation test relation for relaxation test from initial total strain of 0.00172.

AUXILIARY CREEP TESTS ON CHROME MOLYBDENUM STEEL AT 485° C.

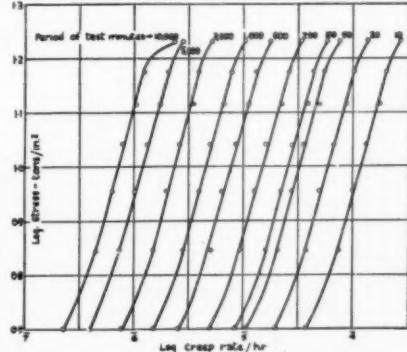


TABLE I.—RELAXATION TESTS ON 0.17% C STEEL AT 455° C. RECOVERY* STRAINS OCCURRING IN RELAXATION TEST FROM 0.00588 INITIAL TOTAL STRAIN.

Relaxed Stress:— 12.7 to 9.02 tons/ sq. in.		Relaxed Stress:— 9.02 to 8.02 tons/ sq. in.		Relaxed Stress:— 8.02 to 7.58 tons/ sq. in.		Relaxed Stress:— 7.58 to 6.96 tons/ sq. in.		Relaxed Stress:— 6.96 to 6.475 tons/ sq. in.	
Period of Relaxation Test:—8.5 min.		Period of Relaxation Test:—133 min.		Period of Relaxation Test:—557 min.		Period of Relaxation Test:—3267 min.		Period of Relaxation Test:—13367 min.	
Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶
0	328	0	88.1	0	38.6	0	54.8	0	42.7
0.5	331	12	90.7	5	39.3	1	55.5	18	43.9
4.5	335	19	92.0	10	39.9	20	56.7	48	44.8
12	336	34	92.5	25	40.3	60	58.2	73	45.2
50	338	104	94.0	925	42.6	70	59.4	173	46.0
80	339	149	95.1	1010	42.5	449	59.8	278	46.7
230	341	239	95.9	1373	42.3	479	60.2	373	46.7
395	341	329	96.0	2380	42.4	1479	61.3	388	47.8
1415	339	1289	96.7	2813	42.4	1988	61.5	628	47.6
1575	338	1739	95.9	3820	42.7	2699	62.2	808	48.0
1881	339	2729	95.8	5693	42.6	3359	62.3	1858	48.3
2405	338	3179	96.5	6700	42.7	4139	62.3	3268	48.5
2855	337	4259	96.0	9580	42.8	5669	62.5	3688	48.7
5740	333	4619	96.0			7019	62.2	4678	48.4
Recovery	5529	5629	96.3			7055	61.8	4798	48.8
	6969	6969	95.9					6568	49.1
	9839	9839	96.6					7558	49.1
	9864	9864	96.2						

* Including in all cases initial elastic unloading decrements.

chrome molybdenum steel test from an initial total strain of 0.0005 the average correction is 11.5%, while in the test from an initial total strain of 0.00172, the correction is 11%. These figures are virtually identical, although both are somewhat higher than those of the carbon steel. Evidently, therefore, in all cases the correction due to recovery strain is of much the same general order.

It is now necessary to examine how far the modification of relaxation creep rates by the recovery correction explains any discrepancy between the experimentally measured creep rates

and those based on auxiliary creep test results, on assumption of either the time hardening or the strain hardening theories of creep.

The following comparison of experimental values of creep rates measured in relaxation tests, uncorrected and corrected for creep recovery, with creep rates corresponding to stresses equal to the relaxed stresses at specific periods in the relaxation tests and computed from the results of auxiliary creep tests on the assumption of the respective time hardening and strain hardening theories, gives some idea of the extent to which allowance for the

recovery correction does or does not bridge the gap between the theoretical estimates of the relaxation test creep rates and the corresponding experimental values.

The comparison in question is shown in Tables VII and VIII. The creep rates derived on the assumption of the time hardening theory and the strain hardening theory respectively, are derived by means of the procedure described below. In Fig. 6 (and similar figures not shown), creep rates obtained from the auxiliary tensile creep tests are plotted against log stress for specific periods. If now in any particular relaxation test the relaxed stress corresponding to a particular period be ascertained, and from Fig. 6, etc., the creep rate corresponding to this stress at this specified period be noted, then this creep rate is that which would occur at that period in the relaxation test were the theory of time hardening to hold.

Similarly for the auxiliary creep tests in Fig. 8 (and similar figures not shown), log creep rate is plotted against log stress for a series of specific plastic strains corresponding to those occurring in the relaxation tests. The method of derivation of Fig. 8, etc., is indicated by Fig. 7. Evidently, if the relaxed stress in a relaxation test

RELAXATION TEST ON 0.17% C STEEL AT 455° C.

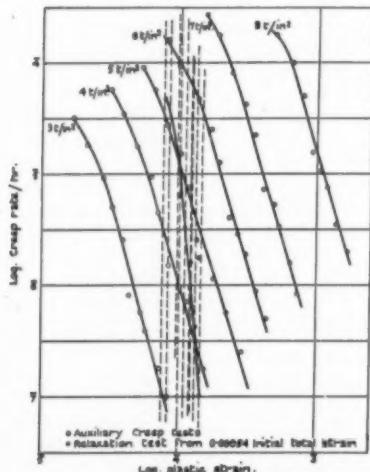


Fig. 7.—Creep rate - plastic strain relations for auxiliary creep and relaxation tests.

TABLE II.—RELAXATION TESTS ON 0.17% C STEEL AT 455° C. RECOVERY* STRAINS OCCURRING IN RELAXATION TEST FROM 0.00054 INITIAL TOTAL STRAIN.

Relaxed Stress:— 5.6 to 5.28 tons/sq. in.		Relaxed Stress:— 5.28 to 4.96 tons/sq. in.		Relaxed Stress:— 4.96 to 4.54 tons/sq. in.		Relaxed Stress:— 4.54 to 4.38 tons/sq. in.	
Period of Relaxation Test:—102 mins.		Period of Relaxation Test:—1487 mins.		Period of Relaxation Test:—1437 mins.		Period of Relaxation Test:—10689 mins.	
Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶
0	34.9	0	27.1	0	37.1	0	14.7
1	35.6	18	27.2	30	39.5	267	16.1
5	36.2	134	27.7	60	39.9	462	16.2
10	36.4	1633	27.7	85	40.2	1437	16.6 ^b
30	36.3	1728	27.8	205	40.4	2875	16.9
75	36.5	2754	28.0	250	41.2	7200	17.2
1930	36.9	2880	28.1	307	41.3	8635	17.4
2860	36.5	5634	27.7	360	41.7		
3310	36.8	7074	28.0	380	41.7		
4300	36.7	7554	28.0	430	41.8		
1750	36.4	8514	28.1	1400	42.2		
5740	36.4	9954	28.5	1540	42.4		
6190	36.5	9974	28.3	4555	42.7		
7180	36.4	10000	28.4	4730	42.8		
Recovery				5720	42.5		
				6170	42.2		
				7205	42.8		

* Including in all cases initial elastic unloading decrements.

TABLE III.—RELAXATION TESTS ON CHROME MOLYBDENUM STEEL AT 485° C. RECOVERY^o STRAINS OCCURRING IN RELAXATION TEST FROM 0-00172 INITIAL TOTAL STRAIN.

Relaxed Stress:— 19.3 to 18.27 tons/sq. in. Period of Relaxation Test:—1 min.		Relaxed Stress:— 18.27 to 17.30 tons/sq. in. Period of Relaxation Test:—55 mins.		Relaxed Stress:— 17.30 to 16.33 tons/sq. in. Period of Relaxation Test:—309 mins.		Relaxed Stress:— 16.33 to 14.48 tons/sq. in. Period of Relaxation Test:—1372 mins.		Relaxed Stress:— 14.48 to 13.48 tons/sq. in. Period of Relaxation Test:—1812 mins.		Relaxed Stress:— 13.48 to 12.35 tons/sq. in. Period of Relaxation Test:—5207 mins.		Relaxed Stress:— 12.35 to 11.52 tons/sq. in. Period of Relaxation Test:—10967 mins.	
Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxa- tion Test × 10 ⁶
0	83.3	0	84.7	0	82.2	0	157.6	0	86.3	0	98.2	0	71.3
0.5	84.8	0.5	86.7	0.5	83.2	0.5	160.4	90.0	109.4	60	108.5	285	86.3
1.5	85.7	1.5	88.3	2	85.3	1.5	163.6	1020	109.1	1545	123.0	465	87.5
2.0	87.0	4.5	91.2	9	89.8	5.5	170.7	1170	108.7	2820	126.2	1455	91.3
2.5	87.9	9.5	93.2	16	91.2	7.5	172.7	1425	109.5	3270	126.1	1945	92.3
3.5	88.8	15.5	94.1	46	93.7	11.5	175.1	2385	110.0	4260	126.3	2895	93.2
4.5	89.7	31.5	95.2	54	94.2	74.5	188.3	2850	110.2	4710	127.3	3345	93.5
6.5	90.8	44.5	95.3	99	96.1	1510	190.0	3840	110.0	5700	127.6	4355	94.3
9	91.3	74.5	96.5	119	96.3	2875	191.8	3930	110.4	6150	128.0	4745	95.1
12	91.8	94.5	96.6	134	96.2	3315	192.0	4005	110.4	7140	129.0	5775	95.4
17	92.5	120	97.3	1089	99.2	3765	191.8	3280	111.3	6225			
37	93.1	145	97.3	1244	99.4	4755	193.5	8130	111.7	7215			
47	93.3	170	97.3	2554	99.8	5205	192.8	9405	111.9				
132	93.4	190	97.5	3979	99.9	6195	192.5						
1086	93.4	1145	98.2	4014	100.1	6645	193.5						
1191	95.3	1405	97.7	4444	100.2	7635	193.1						
1356	95.7	1620	97.8	5424	100.6	8085	193.9						
1551	95.7	2600	98.3	5884	101.4	9175	194.5						
2522	95.1	2750	98.1	6964	100.5	10580	194.5						
2991	95.3	3050	98.2	7424	100.6								
3977	92.8	4088	98.3	8404	100.9								
4431	93.4	4490	98.2	8864	101.0								
5417	93.4	5528	98.1	9839	101.1								
6947	93.1												
8527	93.3												
Recovery	Complete												

^o Including in all cases initial elastic unloading decrements.

corresponding to a specific plastic strain be ascertained, and if the creep rate corresponding to this stress and specific plastic strain be noted in such figures as Fig. 8 corresponding to the auxiliary creep tests, then this creep rate is that which would occur in the relaxation test were the theory of strain hardening to hold.

Owing to the difficulty of accurately measuring creep rates in the auxiliary creep tests at periods less than 10 minutes, the creep rates derived on the bases of the auxiliary creep tests and strain hardening and time harden-

ing theories apply only to periods of 10 minutes and over.

Considering firstly the figures given in Table VII, for the 0.17% C. steel. In column 5 the actual experimentally measured creep rates for the relaxation test are given, while in column 6 the experimental creep rates corrected for creep recovery. In columns 7 and 8 the values based on the auxiliary creep tests and the assumption of either the time or strain hardening theories are given. In the case of the low initial total strain of 0.00054, it will be observed that values based on time

hardening theory are at most periods roughly double those of the (recovery) corrected experimental rates, while the values based on the strain hardening theory are, except for one or two of the short test period values, considerably greater than the corrected experimental values. For the relatively high initial total strain of 0.00588, the

AUXILIARY CREEP TESTS ON 0.17% C STEEL AT 455° C.

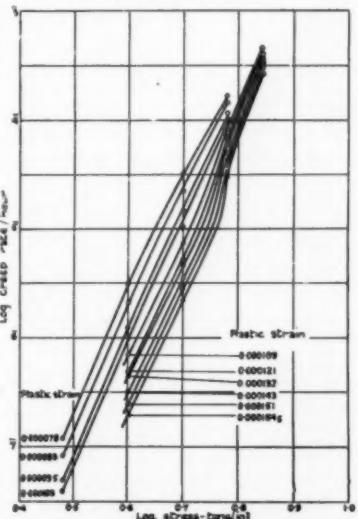


Fig. 8.—Creep rate - stress relations for specific plastic strains corresponding with initial total strain of 0.00054 in relaxation test.

TABLE IV.—RELAXATION TESTS ON CHROME MOLYBDENUM STEEL AT 485° C. RECOVERY^o STRAINS OCCURRING IN RELAXATION TEST FROM 0-0005 INITIAL TOTAL STRAIN.

Relaxed Stress:— 6.10 to 5.66 tons/sq. in. Period of Relaxation Test:—200 mins.		Relaxed Stress:— 5.66 to 5.22 tons/sq. in. Period of Relaxation Test:—1825 mins.		Relaxed Stress:— 5.22 to 4.56 tons/sq. in. Period of Relaxation Test:—5450 mins.		Relaxed Stress:— 4.56 to 4.32 tons/sq. in. Period of Relaxation Test:—13230 mins.	
Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶	Period of Recovery Mins.	Total Recovery due to Stress Decrement in Relaxation Test × 10 ⁶
0	37.5	0	37.2	0	61.7	0	21.2
960	38.8	10	36.9	35	66.7	23	20.8
1440	39.3	130	38.8	350	68.9	110	21.7
2400	39.6	955	40.5	410	70.1	170	22.1
5280	39.6	3865	42.2	485	70.8	265	22.7
5595	41.2	4315	42.7	1445	73.7	1295	23.7
5740	41.7	5305	39.5	1790	73.7	1430	24.1
6735	43.8	5755	39.5	2885	77.3	3065	24.0
7180	45.5	6745	41.3	3330	77.3	4175	23.1
8175	44.7	7195	40.5	4310	78.2	4655	22.3
10000	45.1	8185	41.4	4445	79.0	5615	23.3
						6096	23.6
						7055	24.5
						7335	22.1
						8495	22.5

^o Including in all cases initial elastic unloading decrements.

TABLE V.—RELAXATION TESTS ON 0.17% C STEEL AT 455°C. CREEP AND RECOVERY STRAINS IN RELAXATION TESTS.

Initial Total Strain	Period of Test Mins.	Relaxed Stress Tons/sq. in.	Decrement of Relaxed Stress Tons/sq. in.	Equivalent Creep Strain $\times 10^4$	Total Recovery Strain including Initial Elastic Decrement $\times 10^4$
0.00054	0	5.67	—	—	—
	10	5.24	0.43	38	42
	20	5.18	0.49	43	49
	50	5.05	0.63	55	58
	100	4.96	0.72	63	66
	200	4.89	0.78	69	73
	500	4.75	0.92	81	86 _a
	1000	4.63	1.04	92	98
	2000	4.5	1.17	103	111
	5000	4.4	1.27	112	122
	10000	4.37 _a	1.30	114 _a	126 _a
	—	—	—	—	—
0.00588	0	12.7	—	—	—
	10	9.0	3.7	326	340
	20	8.69	4.01	352	370
	50	8.36	4.34	383	400
	100	8.12	4.58	403	425
	200	7.84	4.86	427	445
	500	7.58	5.12	460	476
	1000	7.34	5.36	472	501
	2000	7.14	5.66	488	523
	5000	6.8	5.91	520	564
	10000	6.6	6.1	537	590
	—	—	—	—	—

values corresponding to time hardening theory are several times the magnitude of those corresponding to the corrected experimental creep rates. No values are given corresponding to the strain hardening theory, as the plastic strain-creep rate curve for the relaxation test lies well beyond the extremities of the auxiliary creep test plots of corresponding nature, for the particular stresses concerned.

Turning now to the figures given in Table VIII and illustrating the case of the chrome molybdenum steel. In the instance of the initial total strain 0.0005, the corrected experimental creep rates are less at short periods and greater at longer periods, than the rates corresponding to the time hardening theory and are at many periods considerably greater than those corresponding to the strain hardening theory.

For the relatively high initial creep strain of 0.00172 the corrected experimental creep rates are lower than those corresponding to time hardening theory, except at the longer periods of test where the rates are not widely different. Rates corresponding to strain hardening are less than the corrected experimental rates.

Reviewing the above remarks, it becomes evident that while in general the experimental corrected creep rates are a closer approach to the values based on time hardening theory than the uncorrected results, they by no means coincide with these values, and may only go a portion of the way towards bridging the gap between experimental and theoretical values.

The corrected experimental values in three of the four cases are more remote from the values corresponding to the theory of strain hardening than the uncorrected values, although the

reverse is true in the fourth case. In no case, however, does any correspondence between experimental corrected creep rates and strain hardening values occur.

Obviously for either of the theories to hold generally complete correspondence with experimental figures is necessary, since partial correspondence in the case of a relaxation test from one particular initial total strain may be accompanied by complete lack of agreement for relaxation tests from other initial total strains.

Apparently, therefore, the factor of creep recovery does not, except in part, explain the discrepancies between experimental relaxation behaviour and theoretical estimates of this behaviour.

TABLE VI.—RELAXATION TESTS ON CHROME MOLYBDENUM STEEL AT 455°C. CREEP AND RECOVERY STRAINS IN RELAXATION TESTS.

Initial Total Strain	Period of Test Mins.	Relaxed Stress Tons/sq. in.	Decrement of Relaxed Stress Tons/sq. in.	Equivalent Creep Strain $\times 10$	Total Recovery Strain including Initial Elastic Decrement $\times 10^4$
0.0005	0	6.10	—	—	—
	10	5.97	0.13	11.6	15
	20	5.91 _a	0.18 _a	16.4	18
	50	5.81	0.29	25.8	27
	100	5.73	0.37	32.8	33
	200	5.69	0.41	36.5	41
	500	5.55	0.55	48.8	55
	1000	5.36	0.74	65.7	71
	2000	5.1	1.00	89	95
	5000	4.48	1.62	144	144
	10000	4.36	1.74	155	182
	—	—	—	—	—
0.00172	0	19.3	—	—	—
	10	18.0	1.3	115.5	130
	20	17.7	1.6	142	160
	50	17.33	1.97	175	185
	100	16.98	2.32	206	223
	200	16.54	2.76	245	267
	500	15.6	3.7	329	350
	1000	14.5	4.8	427	454
	2000	13.42	5.88	523	593
	5000	12.39	6.91	613	720
	10000	11.6	7.7	683	820
	—	—	—	—	—

TABLE VII.—RELAXATION TESTS ON 0.17% CARBON STEEL AT 455°C. COMPARISON OF VALUES OF CREEP RATE MEASURED IN RELAXATION TESTS BOTH UN-CORRECTED AND CORRECTED FOR CREEP RECOVERY, WITH VALUES COMPUTED FROM AUXILIARY CREEP TESTS ON THE BASIS OF TIME HARDENING AND STRAIN HARDENING THEORY.

Initial Total Strain	Period of Test Mins.	Relaxed Stress Tons/sq. in.	Total Plastic Strain $\times 10^4$	Experimental Creep Rates Uncorrected for Creep Recovery $\times 10^4$	Experimental Creep Rates Corrected for Creep Recovery $\times 10^4$	Creep Rates Based on Auxiliary Creep Tests and Time Hardening Theory $\times 10^4$	Creep Rates Based on Auxiliary Creep Tests and Strain Hardening Theory $\times 10^4$
0.00054	0	5.67 (Initial)	—	—	—	—	—
	10	5.24	78	45	49	107	54
	20	5.18	83	25	27	60	30
	50	5.04 _a	95	14	15	29	15
	100	4.96 _a	103	6.6	7	16	10
	200	4.89	109	3.3	3.5	7.4	6.3
	500	4.75	121	2.06	2.2	2.2	3
	1000	4.63	132	0.98	1.05	1.4	2
	2000	4.50	143	0.49	0.53	0.9	1.1
	5000	4.40	152	0.09	0.10	0.39	0.65
	10000	4.37 _a	154 _a	0.038	0.04	0.21	0.45
	—	—	—	—	—	—	—
0.00588	0	12.7 (Initial)	—	—	—	—	—
	10	9.0	5058	149	155	1300	—
	20	8.69	5084	101	105	630	—
	50	8.36	5115	48	51	250	—
	100	8.12	5135	19	20.2	110	—
	200	7.84	5150	9.4	10	43	—
	500	7.58	5182	4.2	4.5	11	—
	1000	7.34	5204	1.95	2.1	6.6	—
	2000	7.14	5220	0.97	1.04	3.7	—
	5000	6.8	5252	0.37	0.4	1.3	—
	10000	6.6	5269	0.16	0.16	0.69	—
	—	—	—	—	—	—	Plastic Strain-creep rate plot for relaxation tests well beyond the extremities of the corresponding plots for auxiliary tests at these relaxation stresses. Strain hardening theory gives results much too low.

Conclusions

(1) For relaxation tests on a 0.17% C. steel at 455°C., and for fairly large and small initial total strains, the correction to be applied to observed creep rates for the effects of creep recovery is only of order 7%. Again, for similar tests on a chrome molybdenum steel at 485°C., and similarly large and small initial total strains, the correction is of order 11%.

(2) For the above two materials under the experimental circumstances noted above, modification of experimental creep rates measured in the relaxation test for the effect of creep recovery does not bring the values of these rates into coincidence with those computed from the results of auxiliary tensile creep tests on the basis of either the time hardening or the strain hardening theories of plastic strain.

(3) It is evident that the factor of creep recovery does not explain the disagreement between experimental behaviour and that deduced from the usual theories; evidently the true mechanism of strain hardening in the relaxation and creep tests has yet to be discovered.

Acknowledgments

The work described above was carried out in the Engineering Division of the National Physical Laboratory as part of the programme of the Mechanical Engineering Research Board and this paper is published by

TABLE VIII.—RELAXATION TESTS ON CHROME MOLYBDENUM STEEL AT 485°C. COMPARISON OF VALUES OF CREEP RATE MEASURED IN RELAXATION TESTS BOTH UNCORRECTED AND CORRECTED FOR CREEP RECOVERY WITH VALUES COMPUTED FROM AUXILIARY CREEP TESTS ON THE BASIS OF TIME HARDENING AND STRAIN HARDENING THEORY.

Initial Total Strain	Period of Test Mins.	Relaxed Stress Tons/sq. in.	Total Plastic Strain $\times 10^4$	Experimental Creep Rates Uncorrected for Creep Recovery $\times 10^8$	Experimental Creep Rates Corrected for Creep Recovery $\times 10^8$	Creep Rates Based on Auxiliary Creep Tests and Time Hardening Theory $\times 10^8$	Creep Rates Based on Auxiliary Creep Tests and Strain Hardening Theory $\times 10^8$
0.0005	0	6.1 (Initial)	—	—	—	—	—
	10	5.97	40	35	40	57.5	16
	20	5.92	45	22	25	29	13
	50	5.81	54	11.5	13	15	9.6
	100	5.73	61	5.3	5.7	12	7.9
	200	5.69	65	2.9	3.2	6.6	6.8
	500	5.55	77	2.2	2.4	3.4	5.1
	1000	5.36	94	1.5	1.6	1.8	3.0
	2000	5.1	117	1.1	1.2	0.79	0.76
	5000	4.48	172	0.78	0.9	0.3	0.1
	10000	4.36	183	0.21	0.24	0.16	0.1
0.00172	0	19.3 (Initial)	—	—	—	—	—
	10	18.0	211	197	216	270	78
	20	17.7	237	99	108	140	65
	50	17.33	270	52	56	74	52
	100	16.98	301	28	31	52.5	40
	200	16.54	340	20	22.2	32	25
	500	15.6	424	15	16.2	14	12.6
	1000	14.5	522	8.1	8.8	7.6	4
	2000	13.12	618	5.2	6.8	3.5	1.7
	5000	12.4	708	4.4	4.6	1.6	0.93
	10000	11.6	778	0.68	0.8	0.9	0.63

permission of the Director of the Mechanical Engineering Research Organisation and the Director of the National Physical Laboratory.

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THE Mond Nickel Fellowships Committee invites applications for the award of Mond Nickel Fellowships for the year 1949. Awards will be made to selected applicants of British nationality educated to University degree or similar standard, though not necessarily qualified in metallurgy, who wish to undergo a programme of training in industrial establishments; they will normally take the form of travelling Fellowships: awards for training at Universities may be made in special circumstances. There are no age limits though awards will seldom be given to persons over 35 years of age. Each Fellowship will occupy one full working year. The Committee hope to award up to five Fellowships each year of an average value of £750 each.

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U.K. Lead and Zinc Price Decrease

The Ministry of Supply announces that its selling prices of virgin lead and zinc are reduced by £17 and £5 per ton respectively with effect from 4th April.

Hot-Dip Galvanising

A Practical Technique For Hydrochloric Acid Pickle Control By Thomas B. Crow, * Ph.D., F.R.I.C.

The operation of pickling steel components prior to hot-galvanising is intended to clean the surface to be treated and assist bonding so that the spelter provides a high degree of protection to the base material. In this operation, strength and temperature of the acid bath, duration of immersion, and the maintenance of suitable operating conditions, are important factors and involve the need for control to achieve the desired result at a reasonable cost. This Report is concerned with an investigation carried out from the initial starting up of a hot-galvanising plant and deals with the development of a technique for the proper conduct of the pickling operations. The technique described, although primarily for the treatment of steel window frames, is generally applicable in the ferrous industry.

Introduction

DURING the last twelve months or more, the author has been giving some attention to the installation and initial starting-up of a hot-dip galvanising plant primarily intended for the rust-proofing of steel window frames. In this connection, he has been concerned, amongst other things, with the development of a technique for the proper conduct of the pickling operations. It will be at once apparent that this development involves the transposition of the elementary "text-book" properties of hydrochloric acid and of the known facts of its behaviour towards scale-covered steel from the "test-tube atmosphere" of the laboratory to the more prosaic and commercially practical outlook of the galvanising shop manager. It is thought, therefore, that some account of the progress already made towards this end may be of special value to the managerial staff and to the senior operatives of a firm similarly pre-occupied. It should be noted that the scope of this paper is limited to pickle control; although it has been written primarily for galvanisers, the technique described is of general applicability in the ferrous industry.

Purpose of Pickling

The purpose of pickling is to bring about such a change in the condition of the surface of the mild steel article to be galvanised as will enable the molten zinc to "take" to that surface; that is, to "adhere" or "bond" to it in such a manner that a high degree of protection against certain kinds of corrosive attack is obtained. For the purpose of the present account, pickling may be defined as the immersion of the article for a certain length of time into a bath

of hydrochloric acid¹ which is maintained at a suitable temperature and in such a condition of activity whereby the desired result is achieved at a reasonable cost.

Hydrochloric Acid

Almost every schoolboy knows that the two elemental gases hydrogen and chlorine can combine together to produce a third gas to which the name *hydrogen chloride* is given; this name will be adhered to throughout the present article, when reference to the *gas* only is intended. This gas is rather heavier than air, but is, of course, several hundreds of times lighter than an equal volume of liquid water; an actual comparison of the specific gravities of these three substances being:—

Air	1	(See
Hydrogen chloride	1½	Note 2.)
Water (liquid)	770	

Expressed in another way, it can be said that a pint of liquid water would weigh roughly 600 times as much as a pint of hydrogen chloride. This is an important fact which has a bearing upon subsequent parts of this article.

Hydrogen chloride can dissolve with extreme readiness in water; in fact, under certain conditions, one cub. ft. of water can take up as much as 500 cub. ft. of the gas; or, expressed in terms of weight, 1,000 lbs. of water can take up over 800 lbs. of hydrogen chloride. A solution of hydrogen chloride in water is always known in the laboratory as *hydrochloric acid*, no matter whether it be a weak or a strong solution, but this name is more or less limited in commerce to the very

strong solutions supplied by the manufacturers in glass bottles, carboys or tank wagons. In this article, the expression "conc. hydrochloric acid" means the highly concentrated (strong) solution normally delivered by the manufacturer; if the abbreviation "conc." is omitted, a weaker solution is meant. Such a solution would usually be made by dilution of the conc. hydrochloric acid by the consumer after delivery.

Strength of Hydrochloric Acid

The properties of hydrochloric acid—that is its power to do certain things—depend very largely upon its strength; that is to say upon *how much* of the solution is water and how much of it is hydrogen chloride dissolved in that water; in order to use hydrochloric acid to the greatest advantage, it is necessary to know something of the method of preparation and evaluation of hydrochloric acid of various strengths.

There are three ways of expressing the strength of hydrochloric acid, namely:—

- (1) Per cent. of hydrogen chloride by weight in the solution.
- (2) Grams of hydrogen chloride per litre of solution.
- (3) Specific gravity of the solution.

Let us suppose that we could start with one litre of water and add to it 30.5 litres of hydrogen chloride gas. Now one litre of water weighs 1,000 grams and 30.5 litres of hydrogen chloride weigh:—

$$30.5 \times 1.64 = 50 \text{ grams (See Note 3.)}$$

That is to say that in 1,050 grams of solution there would be 50 grams of hydrogen chloride. In 100 grams of solution there would therefore be:

$$50 \times \frac{100}{1,050} = 4.75 \text{ grams (See Note 3.)}$$

¹ John Williams & Sons (Cardiff) Ltd.

² The use of sulphuric acid as a pickle is not dealt with in this article, although it is quite common in certain types of plant.

³ The weights of a litre of hydrogen, air and hydrogen chloride respectively, under standard conditions. Temperature and pressure are 0.09, 1.3 and 1.1 grams; that of a litre of water being 1,000 grams. These figures are also correct if read as ounces (A.V.) per cubic foot.

This means that such a solution contains 4½ per cent. by weight of hydrogen chloride. It will be noted that this simple calculation takes no account of volumes; we are simply adding a weight to a weight and calculating what proportion of the whole the added weight is. Suppose now we add a second dose of 50 grams of the gas. Then, calculating as before, we have a strength of

$$100 \times \frac{100}{1,100} = 9.1 \text{ grams}$$

or, 9.1% by weight of the solution is gas. Supposing we now make successive further additions of 50 grams and calculate the results in like manner, as a percentage by weight and that we also construct a graph of such data. A curve will be obtained such as that of Fig. 1, from which it will be seen that successive equal additions by weight do not produce equal rises in per cent. by weight. That is in accordance with an elementary mathematical law and has nothing whatever to do with chemistry or physics. It is well, however, to keep the fact itself in mind.

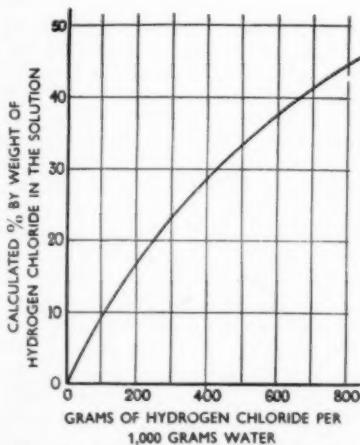


Fig. 1.—Acid strength by weight.

The second method of expressing the strength of hydrochloric acid is to state how many grams of hydrogen chloride are contained in one litre of solution. This, in the author's opinion, is the most easily understood and most practicable of the three methods already mentioned; for it can be easily measured directly in the laboratory; further, the numerical values in grams per litre are also almost exactly

3. A slight departure from strict accuracy in arithmetical calculations or in the stated quantitative physical values will occur from time to time in this article in the interests of simplicity; it has no significance whatever in pickle control.

the same when expressed in ounces (Av.) per cub. ft. (see Note 4). This is a great convenience in converting laboratory test results into useful form in the pickle shop.

If there were no change in bulk when hydrogen chloride is added to water it would be a matter of arithmetic to determine the specific gravity of solutions containing known weights of the gas per given volume of solution. For instance, if 500 grams of gas were added to 1,000 c.c. of water, we should have a volume of 1,000 c.c. and a weight of 1,500 grams, i.e., a specific gravity of 1.5. Actually, however,

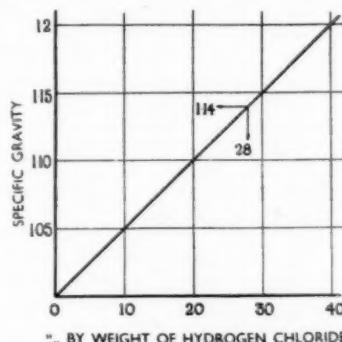


Fig. 2.—Specific gravity in terms of acid strength.

Sp. Gr. of this strength (33½% by weight) is about 1.16. This means that there is an increase of bulk as the hydrogen chloride is added. The assessment of specific gravity in terms of strength must therefore be a matter of direct determination in the laboratory or of calculations derived therefrom. The result of such determination is shown in Fig. 2.

In Table I there are set out the values of the strength of hydrochloric acid (expressed (a) as per cent. by weight, (b) as grams per litre, and (c) as lbs. per cub. ft.), side by side with the corresponding specific gravity. Some published tables also include Twaddell (Tw.) values of specific gravity, but as the present writer does not advocate the use of a hydrometer in a pickle bath at all, they are not included⁴.

For the sake of general interest, the author has included two graphs showing respectively specific gravity *vs* per cent. by weight of hydrogen chloride (Fig. 2) and grams per litre *vs* per cent.

4. Since there are 28 grams to the avoirdupois ounce and twenty-eight litres to the cubic foot, it follows that x grams per litre = x ounces per cubic foot.

5. The conversion of Tw. to Sp. Gr. and reverse is as follows:—

$$\text{Tw.}^{\circ} = (\text{Sp. Gr.} - 1) \times 200. \quad \text{Sp. Gr.} = \frac{\text{Tw.}^{\circ} \times 5}{1,000} + 1$$

TABLE I

Sp. Gr.	Per cent. by Weight (a)	Grams per Litre (b)	Pounds per Cub. Foot (c)	(See below) (d)
1.0032	1	10	.63	30.761
1.0082	2	20	1.26	14.811
1.0181	4	40	2.54	6.836
1.0376	8	83	5.18	2.848
1.0574	12	127	7.92	1.519
1.0675	14	150	9.33	1.139
1.0720 ⁵	14.9 ⁵	160 ⁵	9.98 ⁵	1.000
1.0725 ⁵	15 ⁵	161 ⁵	10.05 ⁵	.987
1.0776	16	172	10.76	.854
1.0980	20	220	13.71	.456
1.1187	24	269	16.76	—
1.1392	28	319	19.91	—
1.1593	32	371	23.16	—
1.1789	36	424	26.49	—
1.1980	40	479	29.92	—

In column (d) is shown the number of volumes of water which must be added to one volume of hydrochloric acid of strength 28° Tw. in order to produce solutions of strengths as expressed in the other columns.

The starred values and all values in column (d) are by the author; others are from "Handbook of Chemistry and Physics" (Chemical Rubber Publishing Company, Cleveland, Ohio, U.S.A.).

by weight (Fig. 3). The first of these is a straight line relation. Another curve of relationship, namely that of specific gravity *vs* grams per litre is not shown, for it would be identical with that of Fig. 3, if in that figure

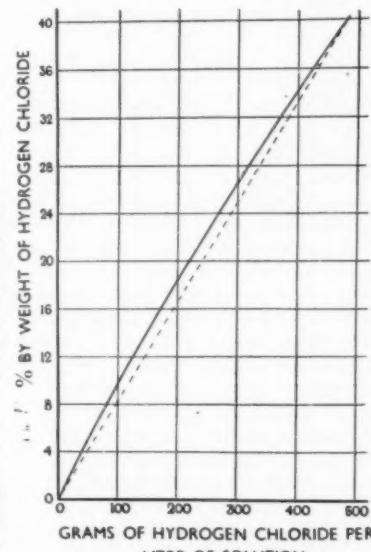


Fig. 3.—Grams per litre *vs* % by weight.

the per cent. by weight values be altered to the corresponding specific gravity values, to which they are strictly proportionate, as already seen from Fig. 2.

There are two other matters affecting the strength of hydrochloric acid which may arise in the pickling shop. If one volume of acid of any strength be added to one volume of acid of any other strength, or to one volume of water, it is safe to assume that the volumes of "mixture" will result⁶.

6. If sulphuric acid and water are mixed in certain proportions, there is a diminution of volume of as much as 8%.

If hydrochloric acid of *any strength* be exposed to the air in any open vessel, such as a pickling tank, it will lose, by evaporation, some water and some hydrogen chloride. Since we are purchasing hydrogen chloride (in the form of hydrochloric acid) for an express and essential purpose, every pound of it which is lost into the air corresponds to an equivalent loss of fulfilment of that purpose and therefore to a loss of money. It is therefore, desirable to attempt to assess the conditions under which the *total* loss will be a minimum from the time when the acid is delivered in concentrated form until it is discharged in the form of spent pickle. It is beyond the scope of the present article to discuss the application of the laws of physical-chemistry to this problem, but the chief factors affecting hydrogen chloride loss are (1) time, (2) atmospheric conditions, including temperature, humidity and freedom from draughts over the tank, and (3) acid or pickle strength. Some of these factors are only partially under control, so that it is not always possible to assess their individual or total quantitative effect. There are, however, a few simple rules which, if followed, will help in minimising hydrogen chloride loss. They are as follows:—

- (1) Storage of concentrated hydrochloric acid must be done in hermetically closed vessels (see No. 4, below).
- (2) If no storage space exists, other than in the open pickling tank itself, dilution of the conc. acid to its working strength should be carried out as soon as delivery is made.
- (3) The total loss of hydrogen chloride from solutions of *any strength* becomes cumulative with time, whether during working or idle periods of an exposed bath. It is therefore, desirable to avoid preparing a fresh bath, even of diluted acid, too far ahead, before it can be put into use.
- (4) Protection, by loose covers, from draughts, during idle periods, reduces loss. If these covers are of heat insulating material, some noticeable conservation of heat also is effected (see Appendix). Loose fitting covers are a poor substitute for a hermetically closed vessel (see (1) above).

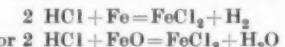
The biggest single factor in causing loss is prolonged exposure of conc. acid. Hence observance of rules (1) and (2) above are the most important.

Provision of covers might be held by some galvanisers to be a rather fussy procedure, but in a very draughty shop in very boisterous weather, it would certainly conserve the strength of an idle bath to some extent.

In regard to dilution, the best procedure is to put the water in the tank first and then pump in the hydrochloric acid by a just submerged hose. By this means a partial mixing is obtained by "specific gravity movement." Thorough mixing, however, will not be complete until several batches of work have been through the bath. It can be assisted by vigorous raising and lowering of the work several times or by propelling it along the bath horizontally, if the tackle permit. Compressed air, bubbled through the bath from a temporarily submerged perforated iron or copper pipe may also be used for agitation, though its prolonged use may seriously weaken the bath, by causing direct loss of hydrogen chloride.

Chemical Action

Hydrogen chloride gas, when dissolved in water, can "attack" iron or oxide of iron (or steel or scale-covered steel) with the formation of iron chloride (ferrous chloride) which remains dissolved in the water. Chemists show this reaction as follows:



and they know that 73 ounces of hydrogen chloride can just consume 56 ounces of iron, forming 127 ounces of ferrous chloride. That last statement cannot be too strongly stressed: one can only get 56 ounces of iron (as iron or iron oxide) dissolved by 73 ounces of hydrogen chloride. If there is an excess of iron, the excess will remain undissolved; if there is an excess of hydrogen chloride, that excess will, likewise, remain unspent. It is important to notice that the *hydrogen chloride* does the work; within certain limits, it does not matter how much water is present; this water can be regarded simply as a carrier or container for the gas and for the resulting ferrous chloride; it is a convenient means of introducing the gas to the article to be pickled.

It would therefore appear that the pickling operation could be worked simply by filling a bath with hydrochloric acid of any strength and immersing batches of iron (steel) articles until all the hydrogen chloride were spent. At that stage the spent pickle could be discharged and could

be replaced by a fresh supply of acid; the cycle would again proceed until exhaustion of the pickle had once more taken place. And so on. This technique might result in chemically successful pickling but it would be slow, inefficient and wasteful. Hence the reason for this investigation.

Bath Control

The general principles in working a pickle bath are (1) to maintain, for as long as possible, a free acid content of about 150 ounces of hydrogen chloride per cub. ft. of carrier (pickle) and (2) to maintain sufficient depth of that carrier to afford total submersion of the work. Let us suppose, for simplicity, that we are working by day only and that we start up in the morning with a bath of clean new acid of "150" strength, properly mixed. During the day there will be a loss of depth of "carrier" due to actual removal of liquid upon the surface of the work; this is known as "drag-out." There will be a genuine consumption of hydrogen chloride in doing useful work and there will be a loss of hydrogen chloride in the drag-out. There will also be losses due to spray, evaporation and other minor causes. At the end of the day, therefore, we shall require to know how much new conc. acid⁷ and how much water we must add to the bath to restore the *depth* to its correct level and also *at the same time* restore the hydrogen chloride strength to the "150" figure, for the next day's work.

The first thing to do, then, when the day's work has ceased, will be to measure the actual lowering of level (drag-out) of the pickle surface. This can be done with a dip stick, or, more conveniently, by a simple device such as is described in an appendix to this article. Since the immersion of work into the bath will cause a rise in the surface level, it is best that depth measurements be always taken when there is no work in it. The next thing to do is to send a sample of the partially spent pickle to the laboratory for a volumetric determination of the free hydrogen chloride in it, in grams per litre (ozs. per cub. ft.). Suppose the drag-out is 3 inches and suppose the gas concentration is down to 130 ozs. per cub. ft. The problem is to add such a quantity of virgin conc. acid and such a quantity of water as will result in a restoration of the level loss, due to the 3 inches drag-out *and a*

⁷ Commercial conc. hydrochloric acid = Sp. Gr. 1.14-28° Tw. and contains about 318 grams. of hydrogen chloride per litre.

acid ; until more This really be d be sence .

working for content oxygen (ickle) with of sersion for day the new e rly ll be e to the n as quine e in ll be the losses other day, now how bath level the the work. when e to the level This more much this work the depth when ing ally r a free per the the ozs. add acid will loss, d a

restoration of strength to 150 ozs. per cub. ft.

"Topping Up"

In the practical application of the method now to be described, the reader is reminded that successful pickle control does not depend upon hair-splitting accuracy of calculation or of depth measurement or of laboratory tests. So long as the bath is replenished reasonably accurately, both in strength and depth, all requirements will have been met.

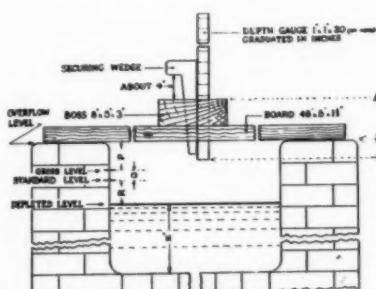


Fig. 4.—Section of a pickling tank.

In Fig. 4 we have a part section of a pickling tank. The "Standard" level is the correct level at the beginning of the day, when the bath is ready to start work, but when there is no work in it. On introducing a batch of work, the surface rises to the "gross" level which will be within a few inches of the "overflow" level, i.e., the top of the brickwork tank. At the end of the day, after removal of the last batch of work, the level has sunk due to drag-out, etc., to the "Depleted" level. The vertical distances between these levels may be denoted by P , Q and R inches, as shown in the figure.

It will be convenient to assume, for present purposes, that a pickle bath of any shape or capacity is made up of a number of adjacent vertical pillars of liquid, each resting upon a base of one foot square. Each foot in depth, therefore, will correspond with a cub. ft. of pickle and each inch in depth to $\frac{1}{12}$ cub. ft.

At the end of the day, let us suppose that the depleted level is x inches and that the strength of the partially spent pickle is y grams per litre (or ozs. per cub. ft.). Let us first see what would be the effect of adding z inches depth of concentrated acid⁷ to the bath in this condition, z , for the moment, being any number of inches.

Then, total ounces of hydrogen chloride in a pillar x inches deep .. = $\frac{x}{12} \times y$

Total ounces of hydrogen chloride in z inches

$$\text{depth of conc. acid} .. = \frac{z}{12} \times 318$$

Adding these together, we have

Total ounces of hydrogen chloride in $(x+z)$ ins.

$$\text{of depth} .. \dots \dots = \frac{xy}{12} + \frac{318z}{12}$$

$$\text{The concentration (C) of this will be} \dots \dots \frac{xy}{12} + \frac{318z}{12} \frac{x+z}{12}$$

That is :

$$C = \frac{xy + 318z}{x+z} \text{ ozs./cub. ft.}$$

This equation, which will be referred to as the "topping up" equation is of perfectly general application to a bath of any size, shape and acid concentration. As will be seen later, it can be used in a variety of ways; when the values of any three of the "unknowns" are known, the fourth can be calculated.

Now, referring to Fig. 4, let us apply this equation to some particular pickle tank. Let us suppose that the standard level is 84 inches deep: that at knocking off time on Monday night, the depleted level is 80 inches ($x=80$) and that the concentration (y) was 141½ ozs. per cub. ft. Suppose now we were to top up to the standard level by adding 4 inches ($z=4$) of concentrated acid. Then, the topping up equation can be worked out with :

$$x=80, y=141\frac{1}{2}, z=4$$

that is

$$C = \frac{(80 \times 141\frac{1}{2}) + (318 \times 4)}{80+4}$$

$$= 150 \text{ ozs. per cub. ft.}$$

That is to say it "comes just right." All that has to be done on Monday night, therefore, is to add 4 inches of conc. acid to the bath and we shall start on Tuesday in order as regards depth and concentration.

Now on Tuesday night suppose

$$x=78, y=142\frac{1}{2}, z=6$$

i.e., that it is decided to add 6 inches of conc. acid to make up the drag-out. Solving the equation, then, with these values, as above, we find

$$C=155 \text{ ozs. per cub. ft.}$$

That does not "come just right";

the bath will be too strong. We will leave it at that for the moment, however, and consider Wednesday evening's conditions. Here we might have

$$x=79, y=130, \text{ and } z=5$$

which gives

$$C=141 \text{ ozs. per cub. ft.}$$

This, too, is not satisfactory.

From a consideration of these three typical examples, it will be clear that all values of strength and drag-out will fall into one or other of three categories, namely :—

- (1) That in which the concentration is correctly restored by restoring the drag-out exactly with concentrated acid.
- (2) That in which the replacing of the whole of the drag-out by conc. acid alone causes an excess of pickle strength.
- (3) That in which the replacing of the drag-out loss with concentrated acid alone still leaves a deficiency of pickle strength.

Category (1) can be left to itself and requires no further discussion. In the cases which fall in category (3) it will be clear that if the bath were of *unlimited depth*, it *would* be possible to add sufficient acid of "318" strength to bring a seriously weakened bath up to "150" strength, and this fact provides an argument (though not a very strong one) for constructing and working the baths with a large "safety" depth margin between the standard and the overflow levels. Other considerations, however, usually prevent this being done and in practice it would not be possible or advisable to raise the standard level much above an inch. As has already been hinted, if restoration of drag-out brought the acid concentration very near to, though less than, the normal 150 figure, the bath could nevertheless be satisfactorily worked without anxiety.

It will be seen that if the conditions of category (3) were to persist day after day, the bath strength will get progressively weaker. One could then, of course, "artificially" bring about a large drag-out by a deliberate partial emptying of the bath and replacing with conc. acid (see Appendix for the relevant calculation) but this procedure would involve the scrapping of an appreciable quantity of good "live" hydrogen chloride which has to be paid for. A decision as to whether this scrapping should be done, or whether the bath should be run to exhaustion point, as described later, would have to be made and such a decision would

TABLE II

When x is ... ins.	and y is ... oz./cub. ft.	and z is 1, 2, 3, 4, 5, 6 inches respectively, the corresponding increase in concentration in oz./cub. ft. will be :—				
72	80	3.26	6.43	9.52	12.53	15.46
	96	3.04	6.00	8.88	11.68	14.42
	112	2.82	5.57	8.24	10.85	13.38
	128	2.61	5.14	7.60	10.00	12.34
	144	2.38	4.70	6.96	9.15	11.30
	160	2.16	4.27	6.32	8.32	10.26
75	80	3.14	6.18	9.15	12.05	14.88
	96	2.93	5.76	8.53	11.23	13.87
	112	2.72	5.34	7.92	10.43	12.88
	128	2.51	4.93	7.31	9.62	11.87
	144	2.29	4.51	6.69	8.82	10.86
	160	2.08	4.10	6.06	8.00	9.87
Etc. (by 3 in.)	Etc.	Etc.	Etc.	Etc.	Etc.	Etc.
	80	2.70	5.34	7.94	10.46	12.93
	96	2.53	4.99	7.41	9.76	12.06
	112	2.35	4.62	6.86	9.06	11.18
	128	2.16	4.27	6.34	8.35	10.32
	144	1.98	3.90	5.81	7.65	9.46
	160	1.79	3.55	5.26	6.94	8.58
						10.19

depend largely upon the iron content (q.v.) of the bath at the time. Other factors affecting the decision might be the non-availability of new acid in sufficient quantity; the possible need for emptying for cleaning or repair; pending shut down for vacation or other purpose.

We now have to consider the remaining set of bath conditions, previously referred to as category (2), in which the restoration of the whole of the drag-out with conc. acid would result in an excess of pickle strength. Such a case will be dealt with by making use of the topping up equation in which z is the unknown. Referring back to the case of Tuesday, in which x was 78 and y was 142½ we have

$$C = \frac{78 \times 142\frac{1}{2} + 318z}{78+z}$$

But C is to be 150, as we wish to restore the bath to this value, Hence

$$150 = \frac{11115 + 318z}{78+z}$$

whence

$$z = 3\frac{1}{2} \text{ inches approximately.}$$

That is, the addition of 3½ inches of conc. acid would restore the strength. But that still leaves 2½ inches of drag-out to be made good. Obviously this will be done with 1½ in. of conc. acid and 1½ in. of water, making total conc. acid 4½ inches.

Rules for Topping Up

The information so far given under this head may be summarised in a series of rules, as follows :—

- (1) Measure the drag-out ($=z$ ins.). From it calculate the depth of the depleted level ($=x$ inches).
- (2) Determine the ozs. per cub. ft. of hydrogen chloride in the pickle ($=y$).
- (3) Apply the topping up equation,

using known values of x , y and z . Calculate C . The value of C will be in one of three categories, as before, namely :—

- (1) 150 (proceed according to 4)
- (2) greater than 150 ... (proceed according to 6)
- (3) less than 150 ... (proceed according to 5)
- (4) Top up with z inches of conc. acid and then proceed according to 9.
- (5) Restore the drag-out with z inches of conc. acid but remember that even after this is done, you are still below strength. This will probably become progressive as the days go on. Consideration may then be given to a partial emptying of the bath (see Appendix). One other course is to allow pickling to proceed to the point of exhaustion.
- (6) Apply the topping up equation, giving the value of 150 to C ; give x and y their known values and thus calculate z . This will be less than the measured drag-out. We will call it z_1 . Subtract z_1 from z . Call the difference D . Proceed according to 7.
- (7) Add together z_1 and half of D . Proceed according to 8.
- (8) Check all calculations. If correct add $(z_1 + D/2)$ inches of conc. acid and $D/2$ inches of water. This restores depth and concentration to their proper values.
- (9) First batch of work following topping up should be raised and lowered several times to assist mixing. After the first batch has been removed from the pickle, a sample should be tested for concentration as a re-assuring check up.

Topping Up Tables

It has already been explained that the topping up equation can be used to find the fourth "unknown" when the other three values are known. Now, suppose we give x and y certain values, and, giving z the six values, in turn, from 1 to 6, we work out the corresponding six values of C . The six answers thus obtained will tell us what will be the effect on the concentration of adding 1, 2, ..., 6 inches respectively of conc. acid, when the depth was x and the concentration y . Now, still keeping x at the same value, increase the value of y by 16 ozs. per cub. ft. and giving z the values 1 to 6 as before, work out the values of C again. These answers will give us the information for the same depth but for a higher concentration.

With sufficient time and patience we could work out the effect of additions of from 1 to 6 inches of conc. acid upon all depths and all concentrations between certain limits. For the sake of illustration, let x be 72, 75, 78, 81, 84, 87; let y be 80, 96, 112, 128, 144, 160; and let z be from 1 to 6.

The results of so comprehensive a system of calculations could be tabulated in some such manner as is partially shown in Table II. Prospective galvanisers, however, are not advised at the outset to embark upon the construction of such a table. When their plant has been in operation for some time—maybe some twelve months—sufficient data ought to be available from the daily records of C , x , y , z to enable the range and increments of these quantities to be fixed. For instance, in the whole of twelve months x may never have varied by more than 6 inches and y by more than between 150 and 120 ozs. per cub. ft. In such a case the values to be assigned to x might be one inch apart whilst those for y could be 5 oz./cub. ft. apart.

Such a table, once made, will give instant information for topping up. For example, suppose it shows that when the drag-out was 5 inches, the addition of 2 inches of conc. acid would restore concentration to 150. This would leave 3 inches still to be made up, obviously with ½ and ½ conc. acid and water; total acid, therefore, will be three and a half inches, water, one and a half. A glance at such a table, moreover, will show under what conditions nothing (short of resort to "artificial drag-out") could be done to restore the strength to its full value.

(To be continued.)

The Tensile Strength of Titanium at Various Temperatures

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An investigation is described which was carried out to determine the effect of temperature on the tensile strength of titanium of known composition.

Introduction

IT is known that titanium of moderate purity has some good mechanical properties at room temperature. Values for the Brinell hardness at temperatures between 210° and 550° C. have been given by Kroll,¹ and he has stated² that vacuum-melted material with a Brinell hardness of 180–200 at room temperature has a hardness of about 87 at 400° C. Apart from his results, however, it was not known to what extent the mechanical properties altered as the temperature was raised, and it was decided, therefore, as part of a programme of work on titanium to prepare a small quantity of metal of known composition, measure its tensile properties and hardness at room temperature, and determine the effect on the tensile strength of raising the temperature of testing to 300° and 500° C.*

Preparation of Specimens

Kroll² has described the preparation of a fairly pure titanium by the reduction of the tetrachloride with magnesium. The magnesium reduction process has also been used by Hanna and Worner³ and has been developed by Dean and co-workers^{4, 5} for the production of a powder from which ductile compacts could readily be made by sintering at a moderate temperature. Since the procedure described by Dean is a relatively simple one and has been shown to be capable of producing material which may be easily worked⁶ and which has good room temperature mechanical properties, it was adopted in the present case. Approximately 100 grams of titanium powder were obtained from one "run" of the reduction apparatus.

The magnesium was melted in a clean mild steel pot and redistilled titanium tetrachloride (the purification of which is discussed by C. K. Stoddard and E. Pietz⁷) was run in from a glass apparatus, the flow being regulated by a well-fitting ground-glass stop-cock lubricated at the edges with a silicone grease. After the reaction had been completed and the pot had been opened, the reaction products were removed by a turning operation on a lathe and leached; the metal was then milled, leached for a second time, separated from material coarser than 36 and finer than 240 mesh B.S.S., washed with water and dried *in vacuo*. A few particles of loose iron too large to be dissolved during leaching were removed magnetically.

Three briquettes of approximate dimensions 3 × $\frac{1}{2}$ × $\frac{1}{2}$ in., each weighing 30 grams, were made using a pressure of 50 tons/sq. in. This pressure was used in

order to make the compacts as dense as possible before sintering. The specimens were sintered together in a vacuum furnace for 16 hours at 1,200° C. Once the occluded hydrogen had been evolved, the pressure was measured by an ionisation gauge and a degree of vacuum was obtained by an oil diffusion pump sufficient to prevent appreciable contamination of the specimens by gases; however, as a precaution against the development of small leaks the specimens were enclosed in a molybdenum sheet container filled with scrap titanium.

In the course of experiments on the preparation of titanium by the iodide process we have observed that thin surface films of oxide disappear by diffusion into the metal after an hour or two at 500°–550° C. *in vacuo*. This confirms previous observations by Fast.⁸ Surface films of nitride also disappear by diffusion, as shown by experiments at somewhat higher temperatures.⁹ The surface film on the particles of the pressed compact can, therefore, be assumed to be without significant influence on the course of the sintering process.

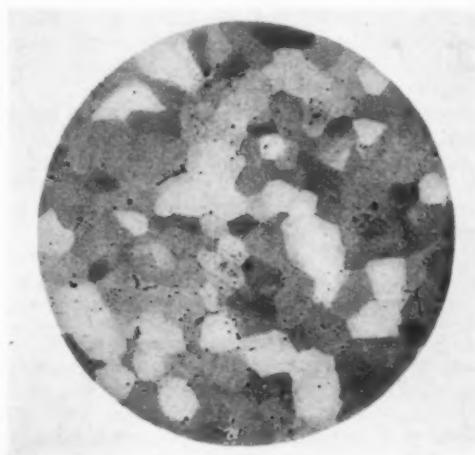


Fig. 1.—Showing the structure of titanium × 100.

After sintering the density was approximately 4.3 grams/cc. The bars were given a small reduction by cold hammering and were heated again to 1,200° C. to allow further sintering to take place and to ensure removal of the last traces of magnesium. They were then cold hammered to a density of 4.49 grams/cc. with two intermediate vacuum anneals at 800° C., and were annealed for 2 hours at 800° C. after the last working operation.

* Since this paper was authorized for publication, C. I. Bradford (*Iron Age*, 1948, Vol. CLXII, No. 27, p.41) has given some similar data, but the composition of his material is not stated.

Microscopic examination revealed the equi-axed structure shown in Fig. 1 (etched with boiling 50% HCl). No significant amount of porosity could be detected, but there was some segregation of impurities in grain boundary regions. No segregation on a macro scale was observed.

Mechanical Tests

Three 0.113 in. diameter sub-standard tensile test-pieces were machined from each bar and were tested at room temperature, 300° and 500° C. The results are given in Table I. The figures quoted are each an average of three experimental results, with the exception of the room temperature elongation figure which is a single result, the other two specimens having broken outside the gauge length. The individual tensile strengths lay within 4% of the values given. One specimen from each bar was tested at each temperature. The tests at the two higher temperatures were carried out according to the procedure laid down in B.S.1094: 1943 (i.e., after having been heated for 1 hour at the testing temperature the specimens were extended at the rate of approximately 0.1 in./in./min.).

TABLE I.—TENSILE TEST RESULTS

	Room temperature	300° C.	500° C.
Proportional limit ..	25.4 tons/sq. in	—	—
0.1% Proof stress ..	33.0	—	—
0.2%	34.3	—	—
0.5%	35.7	—	—
Max. stress	41.1	17.3	12.2
Elongation %	33.2	26.3	16.6
Reduction of area %	41.7	—	—

The Vickers hardness (HD/10) was 227. This falls within the range 225–250 quoted by Campbell and co-workers¹⁰ for annealed titanium made by the Bureau of Mines process but is slightly higher than values given by Kroll.³ As a further comparison the Rockwell A hardness was measured and found to be about 54. This agrees with the figures published by Dean⁴ for annealed material.

Chemical Analysis

Portions of the bars and the tensile test-pieces were analysed and the results are given in Tables II and III.

TABLE II.—CHEMICAL COMPOSITION OF THE TITANIUM.

Element.	Weight per cent.
Titanium	99.68
Oxygen	0.19
Silicon	0.07
Carbon	0.05
Iron	0.05
Aluminium	0.02
Chromium	0.0035
Manganese	0.002
Cobalt	0.001
Nickel	trace

The following elements were not detected:—Nitrogen, magnesium, vanadium, zirconium, tantalum, columbium, zinc, calcium, lead, copper. The metal was not analysed for chlorine, sulphur or hydrogen; but no evidence for the presence of any of these elements was obtained, and it is thought that they can only have been present in very small amounts.

It will be seen that no magnesium was present and that nitrogen, which in small amounts has a more marked influence on the mechanical properties than oxygen,¹¹ was also not detected. It was sought by a micro-Kjeldahl method. The metal might perhaps be expected to contain less nitrogen than oxygen because of the slower rate of diffusion of nitrogen in titanium,¹² and the slower rate of reaction of nitrogen with titanium observed by Carpenter and Reavell.¹³ The results of vacuum fusion analysis in Table III, however, indicate

that there was a small nitrogen content. The figure for the oxygen content given in Table II was obtained by the chlorine method—a method which has been used for the determination of oxygen in zirconium by Lilliendahl, Wroughton and Gregory¹⁴ who analysed zirconium samples to which known amounts of oxygen had been added and showed that reasonably accurate results could be obtained. The value given may have been slightly low on account of the presence of carbon, and a slightly higher figure was obtained by vacuum fusion. Taking the carbon content into account the values are in good agreement.

TABLE III.
THE OXYGEN AND NITROGEN CONTENTS DETERMINED BY VACUUM FUSION.

Oxygen Nitrogen
0.27 ± 0.02% 0.09 ± 0.02%

The amount of oxygen may be in solution since Ehrlich¹⁵ has found the solubility limit to be about 12% of oxygen in the hexagonal phase at a not accurately known temperature in the neighbourhood 750° C.

Discussion

Small quantities of oxygen, nitrogen, and to a lesser extent silicon,¹⁶ are known to cause a marked increase in the room temperature hardness of titanium. Campbell and others¹⁰ have measured the hardness and tensile strength of titanium made by the dissociation of the iodides and have found a Vickers hardness (10 Kg.) of about 90 and a tensile strength of 27 tons/sq. in. A hardness figure of this order has also been found with similar material made in this laboratory. It appears, therefore, that the small quantities of impurities found in the present case have more than doubled the room temperature hardness and increased considerably the tensile strength; and it is possible that the effect may not have been as great as it otherwise would have been owing to the slight micro-segregation which was observed. In spite of the impurity content the metal could be cold worked, though not to the same extent as is possible with iodide material. It is possible, however, to hot work titanium with a considerably higher oxygen content than the material described here.

The rapid drop in hardness with temperature found by Kroll is accompanied by a corresponding drop in tensile strength.

Acknowledgments

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The "Ring Balance" Flow Meter

By S. J. G. Taylor, B.Sc.

The importance of accurate flow measurement in the efficient operation of gas-fired furnaces is emphasised in this article, in which the author considers in some detail the "ring balance" type of flow meter. After discussing the theory and construction of the instrument, he proceeds to deal with some of the practical implications of the theory and finally considers the question of accuracy and sources of error.

THE present urgent need for fuel efficiency emphasises the importance of the measurement of the flow of the reactants to the combustion process; indeed such action is the logical first step to increased efficiency, facilitating as no other method can, the investigations of the engineer, and enabling him to issue precise instructions to operatives, when optimum conditions have been ascertained. A large number of such cases concern the measurement of the flow of low pressure gases, such as air, coke oven gas and blast furnace gas, and the means by which this is achieved is almost always by the installation of a differential pressure device (e.g., orifice plate, venturi tube) in the supply pipe-line. Several types of flow meter operated by differential pressure are in use, one of which is the ring balance meter.

The utility of any measuring instrument is much reduced if its readings are in error, so that it is the duty of the conscientious investigator to spend at least a little time in the study of the working principles of the instruments, by the readings of which he will endeavour to draw conclusions, in order that he may have a knowledge of the likely accuracy of the readings he takes and know how to obtain the maximum precision from the instrument. A knowledge of the theory governing the practical instructions issued by the instrument manufacturers is required for a complete understanding, and in the following account some theoretical considerations which have a bearing on the practical use of the meter are discussed.

The ring balance meter is, in principle, a U-tube manometer, in which a difference in level of the liquid in the arms produces mechanical movement which may be used to move a pointer over a scale, or a pen over a chart, or control a counter mechanism, as in integration of the rate of flow. Just as a U-tube manometer can be used for measuring "static" pressures or draughts, with one limb only connected to the source of pressure or draught and the other limb open to atmosphere, so also could a ring balance meter be used, though instruments operated by diaphragms or bellows are generally cheaper and more compact for this purpose. Therefore, consideration is restricted to the use of the ring balance meter as a means of measuring flow.

Construction

The "ring balance" consists of a hollow round ring which is commonly either of circular or rectangular cross section. The ring is pivoted at its centre of gravity, which should closely correspond with its geometrical centre. A blanking plate is inserted at the top of the ring, and to each side of the blanking plate is connected a flexible pipe which runs to a connecting

head at the meter case. One side of the ring is thus connected to the upstream tapping and the other to the downstream tapping of the differential pressure producer. The lower section of the ring contains a liquid, usually paraffin.

When the differential pressure is disconnected from the ring, and the equalising valve of the meter is open, the manometric liquid is free to flow until its centre of gravity is vertically below the pivot of the ring, irrespective of the position of the ring and since the centre of gravity of the latter is at the pivot (or has been adjusted to be so by balancing weights), the ring should remain stationary in any position within the expected range of movement. If now a differential

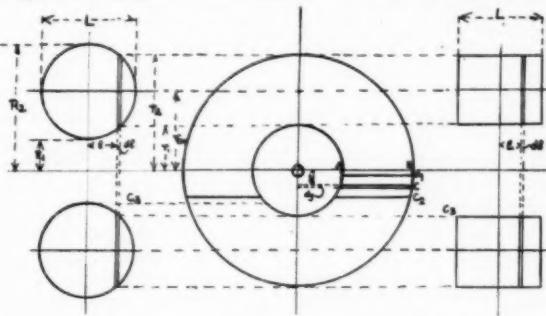


Fig. 1.—
Circular Ring.

Fig. 3.—
Fig. 2.—
Rectangular Ring

pressure is applied so that the pressure is higher on the left-hand side of the ring than the right, then the manometric fluid will flow from left to right until a difference in level sufficient to balance the differential pressure is established. The centre of gravity of the liquid will now have been displaced to the right of the vertical plane containing the pivot, and so the ring will experience a couple tending to make it rotate in a clockwise direction. This tendency is counteracted by a controlling couple produced by either a fixed weight acting over a variable distance, or a variable weight acting over a fixed distance. Rotation of the ring ceases when the two couples are equal. Thus, the deflection of the ring depends in the first instance on the magnitude of the couple due to the displaced liquid.

Theory

The ring is either circular or rectangular in cross section, as illustrated in Figs. 1 and 2. For mathematical purposes, imagine each type to be cut up into a large number of thin slices in vertical planes, at right angles to the axis of rotation. One such slice occurring

at a distance l ft. from the centre line of the ring, and of thickness dl is illustrated in Fig. 3. The inner radius of the slice is r_1 ft., the outer radius is r_2 ft. These remain constant as l varies from

0 to $\pm \frac{L}{2}$ ft. in the case of the rectangular ring, whereas in the case of the circular ring, they vary from R_1 ft. and R_2 ft., when $l = 0$ to r_{av} ft. when $l = \pm \frac{L}{2}$. In

the latter case, the slices under consideration are so thin that the variation in r_1 and r_2 over the thickness of the slice need not be considered.

Suppose that the pressures in the left and right-hand sections of the ring above the liquid surfaces are p_1 and p_2 lb. per sq. ft. respectively. As a consequence, the manometric liquid (density ρ lb. per cu. ft.) adjusts itself until the difference in vertical level between its surfaces (i.e., C_1 C_2) is sufficient to balance $p_1 - p_2$.

The liquid below level C_2 is equally distributed about the vertical plane containing the pivot, and so exerts no turning moment. The liquid between C_1 and C_2 exerts a turning moment about the pivot, tending to cause rotation of the drum in a clockwise direction.

Let the density of the manometric liquid be ρ lb. per cu. ft.

Let horizontal distances from O be denoted by x . The positive direction is to the right.

Let vertical distances from O be denoted by y . The positive direction is downwards from O .

Consider a thin strip of liquid, of thickness dy , at level C .

$$\text{Mass of this strip} = \rho \cdot A \cdot B \cdot dy \cdot dl. \text{ lb.} \\ = \rho (x_2 - x_1) \cdot dy \cdot dl. \text{ lb.}$$

$$\text{Moment of this strip about the axis of rotation through } O = dM = \frac{\rho \cdot (x_2 + x_1) \cdot (x_2 - x_1) \cdot dy \cdot dl. \text{ lb. ft.}}{2} \\ = \frac{\rho}{2} (x_2^2 - x_1^2) \cdot dy \cdot dl. \text{ lb. ft.}$$

Now $x_2^2 + y^2 = r_2^2$ and $x_1^2 + y^2 = r_1^2$, thus :

$$dM = \frac{\rho}{2} (r_2^2 - r_1^2) \cdot dy \cdot dl.$$

Total moment of liquid about the axis of rotation through $O = M$.

$$M = \int_{-\frac{L}{2}}^{\frac{L}{2}} \left\{ \int_{y_1}^{y_2} \frac{\rho}{2} (r_2^2 - r_1^2) \cdot dy \right\} dl. \text{ lb. ft.} \\ = \int_{-\frac{L}{2}}^{\frac{L}{2}} \frac{\rho}{2} (r_2^2 - r_1^2) (y_2 - y_1) dl. \text{ lb. ft.} \\ = \int_{-\frac{L}{2}}^{\frac{L}{2}} \frac{h\rho}{2} (r_2^2 - r_1^2) dl. \text{ lb. ft.}$$

where h = difference in level of liquid surfaces, ft.
For rectangular type rings,

$$M = \frac{h\rho}{2} (r_2^2 - r_1^2) L \text{ lb. ft.}$$

For circular type rings, note that $\frac{r_2 + r_1}{2}$ is a constant, and equal to $(R_2 + R_1)/2$ and $(r_2 - r_1)$ is expressible in terms of L and l . Thus :

$$M = h\rho \frac{(R_2 + R_1)}{2} \int_{-\frac{L}{2}}^{\frac{L}{2}} \sqrt{L^2 - 4l^2} dl$$

Performing the integration :

$$M = \frac{\pi L^2}{8} (R_2 + R_1) \cdot h\rho$$

Therefore for circular type rings :

$$M = \frac{\pi L}{4} \frac{(R_2^2 - R_1^2)}{2} h\rho \text{ lb. ft.} \dots \dots \dots (1)$$

For rectangular type rings (considering radii of R_1 and R_2 for the inner and outer circles).

$$M = L \frac{(R_2^2 - R_1^2)}{2} h\rho \text{ lb. ft.} \dots \dots \dots (2)$$

Practical Aspects

The equations for M demonstrate the following points.

Liquid density.—The value of M for a given differential pressure $(p_1 - p_2)$ is independent of the density of the manometric liquid, as the product $h\rho$ is a constant and equal to $(p_1 - p_2)$. Thus equations (1) and (2) could be re-written :

$$\text{Circular ring } M = \frac{\pi L}{4} \frac{(R_2^2 - R_1^2)}{2} (p_1 - p_2) \text{ lb. ft.} \dots \dots \dots (3)$$

$$\text{Rectangular ring } M = L \frac{(R_2^2 - R_1^2)}{2} (p_1 - p_2) \text{ lb. ft.} \dots \dots \dots (4)$$

Thus the density of the manometric liquid need not be adjusted to a particular value, neither will the calibration of the meter be affected by changes in temperature of the manometric liquid.

Quantity of Liquid.—The frictional force at the pivot of the ring will be related to the overall weight of the ring, fittings and liquid, which should therefore be minimised. For a given ring, the only factors that can be varied are h , ρ , and the quantity of liquid placed in the drum. In Fig. 3 it appears that if the left-hand liquid surface fell below the level through C_3 the ring would become "unsealed" and gas could pass from left to right.

In practice, a blanking plate with an opening in the lower part is often inserted at the lowest point of the ring, so that the left-hand liquid surface could drop below level C_3 without the ring becoming unsealed. Further mathematical analysis of this situation reveals that the turning moment produced by a given difference in liquid levels depends not only on this difference in level, but also on the depression of the level in the left-hand side of the ring below C_3 . Moreover, such analysis shows that the turning moment under these circumstances is always less than that which obtains when the left-hand liquid level is above C_3 for the same applied differential pressure. Consequently sufficient liquid should be put into the ring to enable the left-hand liquid level to be at C_3 when the differential head which will cause the maximum deflection of the meter is

applied. Under these circumstances the turning moment is as given by equations (1) to (4), and the weight of liquid in the ring is minimum.

Further increase in the amount of liquid within the ring merely increases the overall weight without in any way affecting the turning moment.

The advantage of using a liquid of low density is also apparent, as although the weights of various liquids to maintain the difference in level ($C_1 C_2$ in Fig. 3) is approximately constant for a given differential pressure, the weight of liquid below C_3 varies directly with density. Consequently the greater the density the greater the overall weight, and the greater the friction at the pivot. In addition to possessing as low a density as possible, the filling liquid should not attack the material from which the ring is made and should possess neither high viscosity, nor high vapour pressure at the working temperature of the meter. Ordinary paraffin accords to this specification quite well.

Instrument size.—The physical size of the meter is governed by the dimensions of the ring, or on the values of R_1 , R_2 and L . Space is better used by a rectangular ring than a circular one, as the formulae show that for the same values of R_1 , R_2 and L , the turning moment in the latter case is less than that in the former by the factor $\frac{\pi}{4} (.786)$.

The expressions for the turning moments show that M increases with increase in $(R_2^2 - R_1^2)$. However, consideration should also be given to the increase in weight of the filled ring with increase in $(R_2^2 - R_1^2)$ in order to ascertain the true effect of increase of the latter term upon the sensitivity of the instrument. Actually, this factor is not within the control of the owner of the instrument, but it may be of interest to record that, in the case of a rectangular ring of fixed width, assuming a fixed value for R_1 , and various values of R_2 the relationship between overall weight of the ring and liquid and the turning moment for a given differential head is almost linear. Of course, the relationship between M and m for a fixed differential head, R_1 , R_2 , and ρ , but with variable L is entirely linear. In the case of a circular type ring where L depends on R_1 and R_2 , by reason of previous statements in this paragraph, we may say that the relationship between M and m is also practically linear.

Deflection.—The angular deflection of the ring under the turning moment caused by the applied differential pressure is governed by the relationship between the controlling couple and the angular deflection. For example, if the controlling couple is directly proportional to the angular deflection, then equal increases in turning moment will cause equal increases in meter deflection.

The controlling couple can either take the form of a fixed force at a variable distance of action, or a fixed distance of action and a variable force. The force is usually that of gravity on a weight.

Let f represent the controlling force, lb. wt., and x the perpendicular distance between line of action of f and the drum pivot, ft.

Then for a rectangular drum $f(x) = \frac{L}{2} (R_2^2 - R_1^2) h \rho \dots (5)$

The relationship between the differential head h , and the rate of flow q , through a differential pressure producer can be written $q = K_2 \sqrt{h}$, where K_2 is a constant

embodying the physical properties of the differential pressure device, and of the fluid passing through it.

Substitute in (5) $f(x) = \frac{K_1}{K_2} q^2$

Now it is a distinct advantage for a meter to display a straight line relationship between the quantity being measured and the angular deflection. Therefore, there should be a straight-line relationship between \sqrt{f} or \sqrt{x} whichever happens to be the variable, and the angular deflection. This is brought about by either a specially-shaped float working in a mercury bath (fixed x) or a specially-shaped cam from which hangs the fixed control weight (fixed f).

Sources of Error

Tolerances.—As the elimination of all friction from the mechanism is impossible, it will be found that the differential pressure necessary to cause a ring balance meter to take up an assigned reading when moving up from a lower reading is always a little above the differential pressure which will cause the same reading to be obtained when the meter moves down from a higher setting. It is therefore, important to have on hand a set of tolerances by which the importance of such differences may be judged. Ring balance meters are usually tested by means of an inclined tube manometer or a micromanometer. Care must be taken to ascertain that the test instrument contains liquid of the correct specific gravity at the temperature at which the instrument is to be used. Also that the instrument is carefully levelled on a firm surface, and that the tube connecting it to the meter is free from dirt or moisture. The greatest accuracy can usually be obtained by applying differential pressures until the meter registers on each major division from 0 to the maximum and then from maximum to 0, and at each stage, reading off the differential pressure from the manometer. We will consider a meter constructed so that a straight-line relationship exists between the angular deflection and rate-of-flow being measured.

Where θ = angular deflection, K 's are constants, q rate of flow, and h differential head.

On test, when a meter reading of θ was obtained, instead of the differential pressure being h , it was found to be $h + \delta h$, so that were the correct differential head applied, the meter would read $(\theta - \delta \theta)$.

$$\frac{\delta h}{h} = \text{error in } h, \quad \frac{\delta \theta}{\theta} = \text{error in } \theta.$$

To find the relationship between $\frac{\delta h}{h}$ and $\frac{\delta \theta}{\theta}$

If δh and $\delta\theta$ are both small $\delta\theta = \frac{d\theta}{dh} \cdot \delta h$

Differentiating equation 7.

$$\delta\theta = \frac{K_4 \delta h}{2 \cdot a/b} = \frac{\theta \delta h}{2b}$$

$$\text{or } \frac{\delta\theta}{\theta} = \frac{1}{2} \frac{\delta h}{h}$$

Thus a 2% error in applied differential head will give rise to a 1% error in indicated flow. This is a very valuable property associated with the measurement of flow by differential pressure methods. Thus for a particular meter, we may draw up a table showing the major divisions on the meter scale, and against each the correct differential pressure to produce each reading, and in addition a column showing the tolerance permissible in the differential pressure before a 1% (or any desired percentage) error in the indication of the meter is incurred. The tolerances in the present case are 2% of the differential heads. The following table assumes a meter graduated 0 to 100 (linear with respect to flow), requiring 1 in. of water differential pressure to produce the 100 reading.

Meter Reading	Differential Head in. w.g.	Permissible Tolerance in Head in. w.g.
10	0.010	≤ 0.001
20	0.040	≤ 0.001
30	0.090	0.002
40	0.160	0.003
50	0.250	0.005
60	0.360	0.007
70	0.490	0.010
80	0.640	0.013
90	0.810	0.016
100	1.000	0.020

A note should always be added, explaining the meaning of the column headed "tolerance." In the above table, the tolerances are the permissible errors in differential head before a 1% error in the indications of the meter are incurred. Such a column may be compiled for any desired error. The figures set a standard by which results from a manometric test may be judged, and also demonstrate that flow meters of the differential pressure type should be designed to operate in the range above 50% of the maximum reading. 0.003 in. water gauge is a reasonable difference in the "up" and "down" readings during a manometric test showing that such a flow meter would be extremely accurate above 50% of the maximum reading whereas at the 10% reading there is a possibility of a 15% error (of the 10% reading).

Balance.—Let us consider the turning moment, produced by a difference in height of .003 in. between the liquid levels in a rectangular ring of typical size, where $L = 4$ inches, $R_1 = 1\frac{1}{2}$ inches, $R_2 = 6$ inches, filling liquid paraffin, specific gravity 0.8. Substituting these values in formula (2) we obtain for the turning moment, $M = .00489$ lb. ft.

Now, the overall weight of the ring, liquid, control, and balancing weights of such a ring would be of the order of 8 lb. In order to produce a turning moment of .00489 lb. ft., the centre of gravity of the 8 lb. would only need to be .0073 in. away from the pivot. This demonstrates the importance of careful balancing of the ring, and also supplies a further reason for minimising the overall weight of the filled ring (for example, by using the minimum quantity of filling liquid).

Friction.—The above case is that of a relatively large force acting over a small distance. A relatively small force could, of course, have the same effect acting over a large distance. With recording ring balance meters, the frictional force of the pen upon the chart is within this category. Careful attention should be paid to ensuring that the pressure of the pen upon the chart is the lightest possible that will ensure continuous marking over the whole width of the chart.

Some other sources of friction are:—

(1) Pivot not accurately horizontal. The ring will eventually slide in the downwards direction until

a sideways force sufficient to counteract this tendency is built up by contact with the end of the pivot, or some other part of the meter.

(2) Contact of the flexible pipes with each other, or with some other part of the meter.

(3) Friction in link mechanisms connected with the indicating pointer, or integrating mechanism.

Transmission.—Besides considering the effect that errors in the adjustment of the meter can have upon its indications, it is not out of place to mention several ways in which the differential pressure may not be correctly transmitted from the differential producer to the meter, thus causing a correctly adjusted meter to register incorrectly with respect to flow, such as:—

(1) Insufficient attention paid to the standardised details relating to the installation of the differential pressure device, and pressure tappings.

(2) Pressure lines improperly drained, or not periodically blown out to remove dust. Careful attention should be paid to the correct method of installing pressure lines. In cases where the gas being measured is liable to contain dust or suspended matter even in small quantities, provision for periodical blowing out with compressed air is usually well worth while.

(3) When a difference in level exists between the meter and differential device, and there is a chance of temperature variation over the length of the pressure piping it is important that such differences are exactly the same for both pipes, otherwise a false differential head will be applied to the meter by reason of the difference in density of the gas in the two pipelines. Such may be minimised by running both pipelines close together.

Oil Storage Tanks

A 30-ft. mild steel oil storage tank recently delivered to Buckingham Palace is one of four which have been made for the new central heating plant being installed in the Palace. The tanks will be located in an underground chamber in the forecourt of the Palace. They were made by Dowson and Mason Gas Plant Co. Ltd., who, in addition to gas plants and industrial furnaces, have, for many years, made a speciality of the manufacture of mild steel storage tanks. It is noteworthy that this firm has also received an order for six similar tanks for the central heating system of the Houses of Parliament.

Industrial Aylesbury Exhibition

INTERNATIONAL ALLOYS LTD., OF AYLESBURY, participated in an Exhibition designed to demonstrate the importance of that town as a centre to both industry and agriculture. Their stand in the Town Hall showed both the aluminium and magnesium alloys produced by the company and also a wide range of the industrial and domestic articles which are produced by other companies from these materials.

In addition, International Alloys loaned to the Exhibition authorities an aluminium alloy building entitled "Almin Hall" which was used to house the prize stock section of the agricultural exhibition. This building was an Alframe building of the new type with increased headroom, produced by a fellow member of the Almin group—Messrs. Structural and Mechanical Development Engineers Ltd., of Slough.

The Design and Operation of Small Mills for Mixing Powder

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Two sizes of ball mills are described, which have been designed to facilitate metallurgical research by powder metallurgy methods, the larger suitable for mixing samples weighing about 10 gms. and the smaller one for dealing with samples of about 1 gm. Reference is made to work carried out with these small mills.

In the course of metallurgical research the methods of powder metallurgy can frequently be made use of with great advantage for preparing alloy samples, for instance, in the investigation of alloy systems. With the exception of the mixing step this process can be carried out satisfactorily on a very small scale; shaking the sample in a tube or grinding it in a mortar does not result in a homogeneous mix. On the production scale ball mills are normally used for this purpose, but the capacity of 1 litre or greater is often far in excess of that

(ii) Tumbling action. As the speed of rotation is increased the mass is carried further round until the balls and powder tumble down over each other, the powder falling between the interstices of the balls. This gives fairly efficient mixing.

(iii) Cascading action. At higher speeds the charge is thrown from the circumference of the mill and cascades to the lowest point. This also gives efficient mixing conditions.

(iv) At much higher speeds the balls and powder are carried round with the mill so that they remain relatively at rest, without any mixing taking place.

Diagrams illustrating these actions are given in Fig. 1. The speeds at which these various stages occur depend on the number and size of the balls used and the powder to be mixed. Experiments varying these factors have been carried out to determine the optimum mixing conditions for the mills now described.

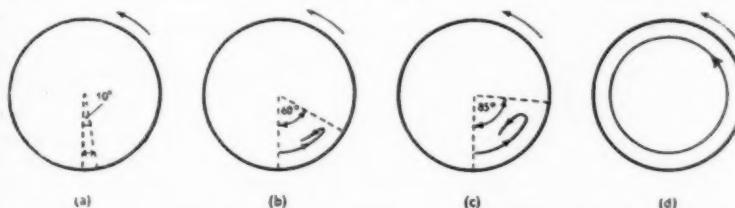


Fig. 1.—Diagram showing different types of movement inside mill at varying speeds. (a) Stick-slip (b) Tumbling (c) Cascading (d) Stage 4 (The angles given are approximate.)

required in the laboratory; we have long used mills of capacities of about 100 cm.³ very successfully with certain metals, but with other softer materials the charge sticks to the mill wall and in any case mills of this size are still larger than are wanted when dealing with rare metals, especially when the material is required for X-ray investigation where only a few hundred milligrammes of sample is necessary.

With this in view, two sizes of ball mills have been designed in this laboratory, in which mixing difficulties have been overcome. The larger mill has a mixing chamber capacity of 75 cm.³ and is suitable for mixing samples weighing about 10 gms., the smaller mill has a capacity of only 10 cm.³ and can deal with samples of about 1 gramme.

Four distinct processes occur within a ball mill, and for a given arrangement are governed by the speed of rotation: in order of increasing speed these processes are:—

(i) A "stick and slip" action where the mass of balls and powder is carried round a short way with the mill until the whole mass slips down to its lowest position, when the process is repeated. This action occurs only at slow speeds and gives only inefficient mixing.

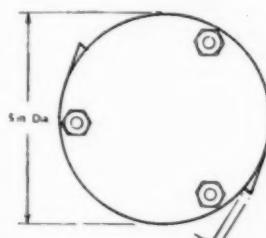


Fig. 2.—75 cm.³ Mill.

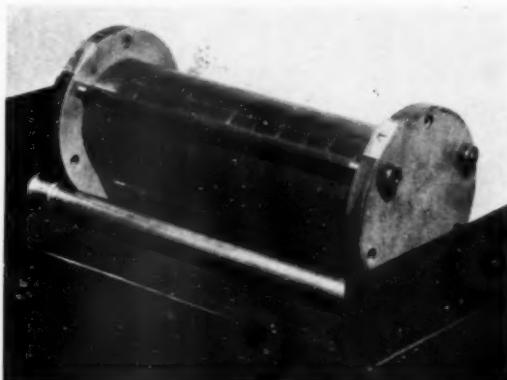


Fig. 3.—Compound 75 cm.³ mill in position on runners.

75 cm.³ Mill

A compound mill consisting of six separate milling chambers is used, Fig. 2. These chambers fit together and are bolted between two 5-in. dia. "Bakelite" end-runners which are fitted with two diametrically opposed brass step insets which cause the mill to drop through $\frac{1}{4}$ in. four times in every revolution. The shock of the drop should eliminate all danger of sticking and aid the mixing process. This compound mill is driven at 60 r.p.m. on two $\frac{3}{4}$ in. dia. steel runners, which are both positively driven by a $\frac{1}{2}$ -h.p. motor with a rotation speed of 1,440 r.p.m., and so spaced that the tangents at the points of contact between mill and runners meet at an angle of 90°-95°. This angle eliminates any danger of the mills jamming between the runners and yet gives sufficiently stable conditions to prevent the mill jumping off during running. This diameter of runner with the 5 in. dia. end pieces of the mill assists considerably in obtaining the desired rotation speed for the mill. A photograph of the mill in position on the runners is given in Fig. 3.

A typical run with these mills is a charge of 10 gms. of mixed powder in each of the six compartments; with 5 gms. of copper and 5 gms. of chromium powder it was found that satisfactory mixing was obtained in twelve hours using either ten to twelve $\frac{1}{2}$ in. or six to eight $\frac{3}{4}$ in. dia. balls; with the latter a certain amount of grinding takes place in addition. Using this "shock" mill no difficulty has been encountered due to the powder sticking.

10 cm.³ Mill

Fig. 4 shows the design of the smaller mill. The milling chamber is made in the shape of a truncated cone so that with the small volume of powder used maximum mixing conditions are obtained. The mill is constructed from mild steel, the two halves bolt together and a thin copper washer fits into a machined recess to prevent leakage of powder when the mill is in operation. Hardened steel bearing balls are used for mixing. The width of a milling chamber must be chosen so that it is not an integral number of diameters of the balls to be used; this is important when the width is small, otherwise the balls may jam. The mill is rotated on a pair of $\frac{3}{4}$ in. dia. steel runners which can be seen in Fig. 5. It was found advisable to have both runners positively driven, and to have the runners separated to give similar contact conditions to those of the mill previously described.

To investigate the mixing conditions a glass plate was fitted over the milling chamber in place of the cover and a series of experimental runs made with a variety of metal powders. In these experiments sticking occurred after a short period of mixing and could not be eliminated either by variation in speed of rotation, sample weight, or the number and size of the balls. Even when the runners of the mill were stepped in a similar way to the previous mill, the sticking still occurred though much longer periods of mixing were required before the trouble was encountered.

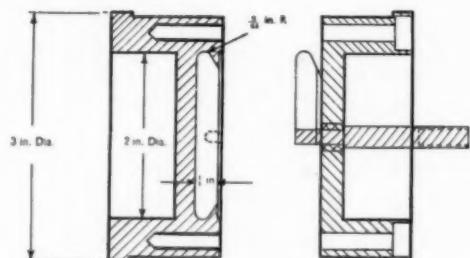


Fig. 4.—10 cm.³ Mill.

To eliminate this the mill was modified, a stationary scraper was fitted so that as the mill rotated it would dislodge any powder adhering to the mill after the cascading action had taken place. The scraper is made of a fairly rigid phosphor bronze strip fitted closely to the contour of the mill chamber. A photograph of the mill with the scraper is given in Fig. 6. The scraper is fixed to a shaft which is keyed externally and is carried in a bronze bearing in the cover of the mill. Experiment with the modified mill showed that sticking troubles had been eliminated, the most satisfactory mixing is obtained using sixteen $\frac{1}{2}$ in. dia. balls and a rotation speed of about 50 r.p.m. When the scraper shaft is lubricated with a thin film of heavy grease there is no trouble through leakage of lubricant into the milling chamber, nor is there any loss of powder along the bearing. Twelve hours mixing with these conditions seems to be adequate to give a homogeneous product. This was verified in a series of alloys of molybdenum and tantalum which were made up by mixing the metal powders in 1 gm.

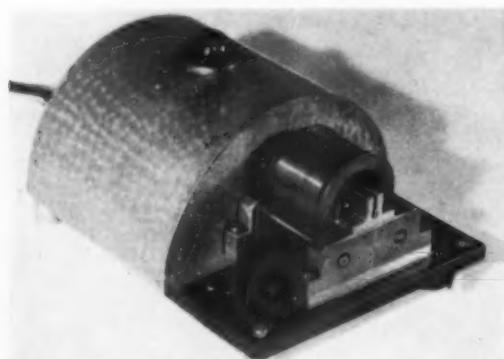


Fig. 5.—10 cm.³ Mill in position on runners.

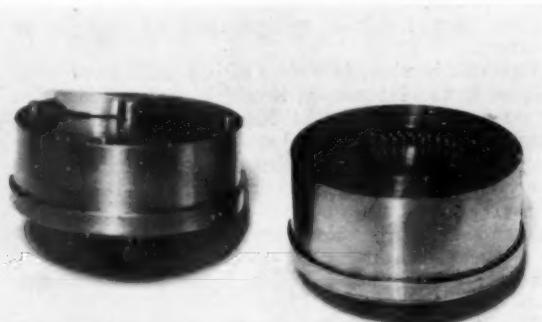


Fig. 6.—10 cm.³ Mill.

samples, pressing the mix to pellets which were sintered at 1,400° C. These two metals are completely inter-soluble and thus measurement of lattice parameter gives an accurate estimation of composition. Different samples from the 70 : 30 Mo : Ta alloy gave good experimental agreement in lattice parameter measurement, showing a homogeneous mixture had been produced.

One such alloy was examined spectroscopically and

showed no trace of copper or iron which might have been picked up from the scraper, mill or balls.

Acknowledgments

The author wishes to record his appreciation of the continued interest in this work shown by Dr. G. A. Geach on whose suggestion it was undertaken, and to express his thanks to Dr. T. E. Allibone, F.R.S. for permission to publish this work.

Welding Can Contribute to National Economy

War-Time Experience Proved it to be Time Saving and Economical

At long last a Government Committee has recommended the increased use of welding as a means of economising steel. In view of the war-time contribution of this process, it is surprising that there should be such a long time-lag in applying it to peace-time industrial activities. Some suggestions are made for its increased application, especially with a view to saving steel.

OPINIONS undoubtedly differ on the extent to which welding can be applied in the various steel consuming industries, but it is worthy of note that the Steel Economy Committee, in their recent interim report, referred to the great importance of welding as a method of economising in the end uses of steel. During the preparation of B.S.449 for steelwork in building the Committee urged the necessity for the incorporation in its rules of design for welded construction which would serve as a basis upon which the more extensive use of welding could take place. The draft code of practice prepared by the British Welding Research Association served as a basis for these design rules in B.S.449 and also for the British Standard for "The Metal Arc Welding of Mild Steel Construction" now in course of preparation.

A committee of members of the Institute of Welding have also prepared an interim report which deals with savings possible by the use of welding instead of riveted construction, and also instead of cast iron where steel fabrications are used as substitutes. This report covers the use of welding over a wide sector of engineering work, including shipbuilding, and indicates, by actual practical examples, considerable weight economies. The examples clearly show that the developments in welding can play a most useful part in the more economical use of available steel, also in enabling entirely new forms of construction to be evolved.

Welding is of great importance from the point of view of stimulating more scientific methods of structural design and by this means forcing a break with obsolete and wasteful practices which are so much part and parcel of the older methods of construction. So far, welding has principally been adopted by the more progressive elements in manufacturing industry and there remains a very large field of steel fabrication where its increased application can lead to most substantial economies in steel.

Applications for welding are so diverse and embrace so many industrial fields that it is not easy to give a general picture of the potentialities of the process for economising

in the use of steel. The difficulty is increased because of the number of methods employed, each of which has its field of usefulness. In the case of arc welding, for instance, growth of its use in this country has been slow in comparison with progress in America. This has been due in no small measure to restrictive tendencies imposed by control. It seemed to be forgotten that the use of arc welding in this country during the war was on a spectacular scale. The reasons for the very considerable increase in welding during that time should have been maintained and should certainly be used as a guide to its greater use now. Its use in naval construction and repair because of the weight of steel saved, its strength, and speed of construction—particularly where the shipyard was suitably laid out to make the best use of welding—made welding of vital importance.

It was especially useful on marine repairs, because arc welding could be carried out afloat and even under water if necessary. Emergency repairs by mobile squads for instance were of particular interest to Mr. P. B. Crighton, managing director of The London Welding Co., Ltd., who was responsible for over 2,000 repairs afloat off the coast of Scotland, when dry docks were full and even wet berths were difficult to find, due to strategic and other considerations, welded repairs afloat filled a vital need in speeding the turn round of ships.

The use of welding in connection with portable military equipment is also well known. Welding lightened heavy army equipment, such as armoured vehicles, gun bases, etc., and its use to replace castings saved many bottlenecks in construction. Its ability to withstand shock was a further great asset. British metallurgists deserve great credit for the rapidity with which they produced the new electrodes called for in such quantities for the welding of high tensile steels and armour plates. It was, however, on bridging sections, light portable pontoons and similar mobile equipment that welding played a part which could not have been filled by any other process. No soldier who went overseas will forget the Bailey Bridge. In the Normandy landings the

quantities of welding on the floating bridges, the shore ramp floats, the unsinkable pier-heads, the towing gear, even of the concrete beetles and floats, was positively incredible.

Mention may also be made of the army welders who fabricated continuous petrol supply lines across North Africa and again across Northern France. PLUTO with its huge conundrums, original in its conception and speedy in its laying of the under Channel section of the petrol supply to France caught the public eye, but the dogged work of the mobile army welding teams which finished only in Germany after crossing hill and valley, river and canal is worth special mention.

In general, welding also played its part in the construction of hangars, bomb stores, sea-plane ramps, underground storage tanks and the like. In fact, there were few uses during the war to which it was *not* put. At the end of the war, along with the natural revulsion to anything warlike, there came, quite inexplicably, a turning away from welding itself. The fall in consumption of electrodes was colossal and those who wanted them found that the raw materials for their manufacture had been restricted. Even if you wanted to weld a ship or a building the electrodes was not available. A fantastic position if ever there were one, conveying no credit to the short-sighted officials responsible. This situation should never have arisen. Welding is vital, at some point, to all our principal industries and not least to the steel industry itself, because, by the reduction in the weight of steel employed in new power stations, factory structures, oil storages, ships, etc., the weight saved by welding would ease the restrictions now imposed and leave more steel for export.

The gradual increase in welded work during 1948 has, we hope, marked the end of the more insensible restrictions on electrodes and an upward trend is now visible in immediate further demands. Welding has played its part, so far as post-war controls have allowed, on useful re-conversion of ships from war service, on chemical plant installations and pressure vessels and on extensive repairs in our gas works all over the country. But welding has not yet touched the fringe of the use to which it should be put on bridges and steel structures. Nor has it been sufficiently applied to reconstructive work. For example, we know that licenses for steel for a new plant or machine to replace an old one may be difficult to obtain, or that delivery of that plant or machine may be so remotely delayed as to make it almost hopeless to order it. There are many cases where a little initiative, re-design or repairs of a few parts, would put the old plant back into effective commission again. The use of welding for "make do and mend" on our older plant and steel structures would be a worth while job in itself.

In view of the recommendations of the Steel Economy Committee it is of interest to consider briefly the application of welding to structural steelwork. The questions most frequently asked regarding it are, is it safe? and is it economical? Both questions are answered in the affirmative, but obviously it is assumed that the work will be done by a reputable structural firm. There are a few occasions where bolted and riveted structures are advisable, but not many.

A welded steel frame building is usually preferred by the architect because it will more readily comply with his outlines and is moreover more functional than a riveted structure could ever be. The factory manager

will prefer it because welded steelwork is not subject to the corrosion problems such as that encountered behind rivet and bolt heads and also because it is easier to apply protective paint. In most cases it gives more headroom and the factory is lighter than when a comparable riveted job is used.

It is probable that the consulting engineer would experience some difficulty in providing the calculations for a multi-storey monolithic steel-framed building and this is a drawback on this type of structure. However, there is no reason to avoid multi-storey construction, since it is quite easy to employ mainly welding and have a limited number of non-rigid connections.

When compound-beams or exceptionally heavily-loaded stanchions are necessary in a building, the simpler welded design will save up to 33% in weight against a comparable riveted design. This percentage is of a high order, but throughout medium and heavy structural work an average saving of 20% is easily made provided the work is designed for welding and not a half-hearted change over in which the basically riveted design is retained, which is by far the commonest mistake made in approaching this subject.

Welded bridges have been successfully completed in various parts of the country and it is time the welded plate girder-bridge, in particular, received more consideration and became more universally adopted than has yet been the case. Such bridges are lighter and more easily maintained.

Another field which offers considerable scope for welding is associated with power stations. Curiously enough some of the most vital parts in this field are welded. Boiler drums and high-pressure pipes are more frequently welded, but strange as it undoubtedly is, the structures themselves contain only a negligible quantity of welding for far greater quantities of steel. An absurd situation if you consider the possible steel saving at a time when so many new stations have to be built, using such enormous tonnages of steel.

At long last a Government Committee has recommended the increased use of welding, but the lethargy which the authorities as a whole have adopted towards welding would be really amusing if it were not so serious in contrast to their repeated cries of "more productivity" and "save steel." After all they have their war-time experience and proof of time saving and economy.

Summary

Steel savings may be effected under the following headings:—

- (1) Ship building—on merchant vessels—warships are already largely welded.
- (2) Structural steelwork.
- (3) Tanks and vessels—although much development has already occurred here.
- (4) Power stations and gas works.
- (5) Railways—bridges and stations—rolling stock is increasingly welded.
- (6) Plant maintenance and reclamation by welding in factories.
- (7) New machinery—weight saved by redesigning from castings to welded fabrications.
- (8) Large steel chimneys, large pipe lines, wind tunnels, crane gantries, excavators, etc.
- (9) Modern design on competitive export markets of fabricated steel of all descriptions.

Dynamic Torsion of Metals and Alloys Used in Aircraft Construction

Their Elastic Limit and Micro-plastic Deformation Under Reversed Torsional Energy Loads—Part III

By Georges Welter

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In modern machinery practically all elements work under short-time repeated loading, and little data is available about the mechanical properties of structural materials investigated under these conditions. The present research has been carried out to investigate the maximum possible impact loads causing no permanent micro-deformation of the test specimen under plus and minus torsion loads.

For the dynamic torsion test two characteristic diagrams are shown, one with a constant hammer weight of 6 lb. and a uniformly increasing dropping height of 1 in. for each dynamic load (Fig. 27a), and one with a constant dropping height of 5 in. and a uniformly increasing hammer weight (Fig. 27b). We also found that for this material very small dynamic loads of the order of magnitude of less than about 8 in.-lb./cu. in. produced a measurable permanent deformation of about 0.00001%. The load-deformation diagram is very steep in this case, and to produce 0.0001% permanent deformation we need about 56 in.-lb./cu. in., and a dynamic load of 90 in.-lb./cu. in. produces a permanent deformation of 0.0005% (curve No. 1). A total preliminary deformation of 0.00144% was produced before reversing the dynamic loads on this specimen. On the other

hand, it appears that this material is very sensitive to stresses beyond the elastic limit in torsion; if dynamically tested in the opposite sense, surprisingly low loads of about 3 in.-lb./cu. in. produce a permanent deformation of 0.0001%; at 9 in.-lb./cu. in. they produce a deformation of 0.00025%; and about 40 in.-lb./cu. in. cause a deformation of 0.001% (curve No. 2). Similar results have been obtained with a specimen loaded under constant speed of 5 in. dropping height. According to Fig. 27b the load-deformation diagram of the virgin material, as shown in curve No. 1, is more or less the same as that given in Fig. 27a (No. 1). This specimen, however, was permanently deformed to a much smaller degree than the first one. Altogether a permanent deformation of about 0.0004% was produced before loading in the opposite sense. Due to a lesser amount of locked-up stresses, the curve

No. 2 (Fig. 27b) is steeper than the one shown in Fig. 27a (No. 2) having suffered a preliminary total permanent deformation of 0.00144%. Thus two to three times higher torsion loads are necessary to produce the same permanent deformation as that for the specimen according to Fig. 27a, No. 1; about 12 in.-lb./cu. in. were necessary to obtain a permanent deformation of 0.0001%; and about 45 in.-lb./cu. in. for a deformation of 0.0004% (Fig. 27b, No. 2).

The effect of the loading speed on this material seems to be less than for most of the other materials tested. For the annealed specimens an increase of only about 8% could be found resulting from an increase of speed from about 48 to over 100 in./sec. (Fig. 28). To produce a permanent set of 0.01%, about 66 in.-lb./cu. in. are necessary under a speed of about 48 in./sec., and only 71 in.-lb./cu. in. at a speed of 100 in./sec.

The effect of Bauschinger as represented in Fig. 29, for a permanent deformation of 0.01%, is, in this case, very great. A preliminary deformation of 0.0001% lowers the dynamic load about 40%, and only 15% of the impact load necessary to deform the virgin material is needed to produce a permanent deformation of 0.0005%. For higher amounts of preliminary deformation surpassing 0.0001%, dynamic loads of only a few in.-lb./cu. in. are needed to produce plastic deformations in this material.

(e) Monel Metal

The test results for this material are represented in Figs. 30-35, of which again the first two Figs. (30 and 31) show the static elastic and micro-plastic load-deformation and energy-deformation diagrams. The static elastic limit of this material is very high

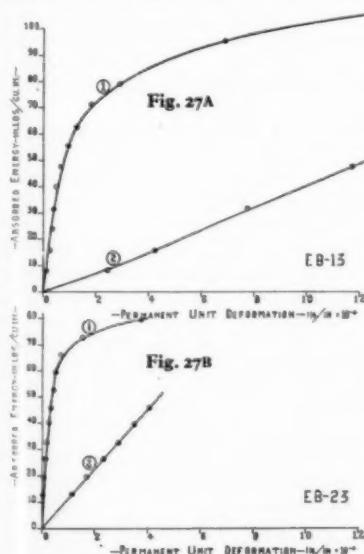


Fig. 27a

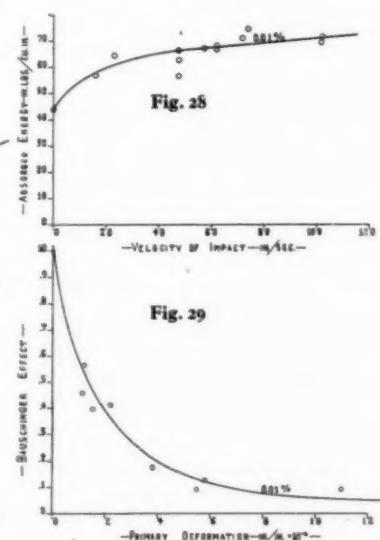


Fig. 27b

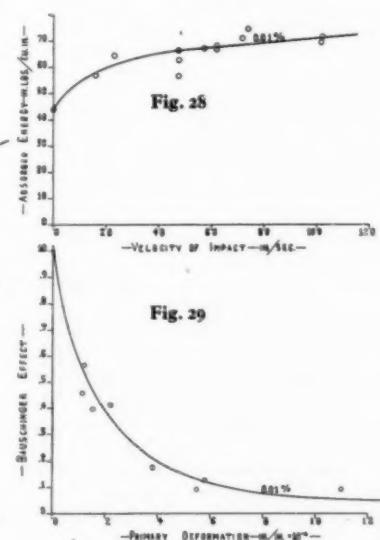


Fig. 28

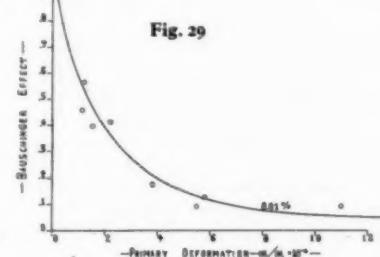


Fig. 29

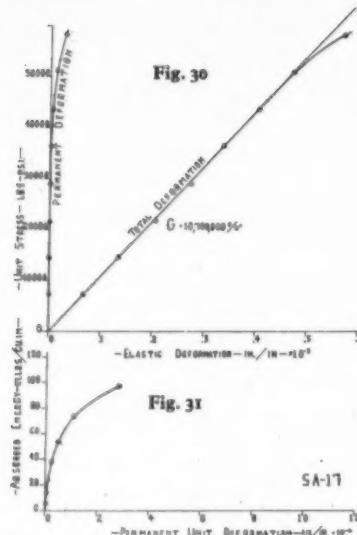


Fig. 30

Fig. 31

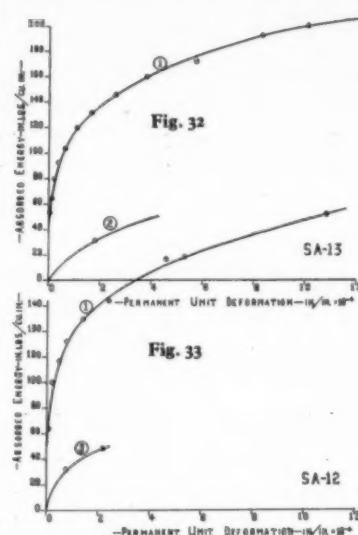


Fig. 32

Fig. 33

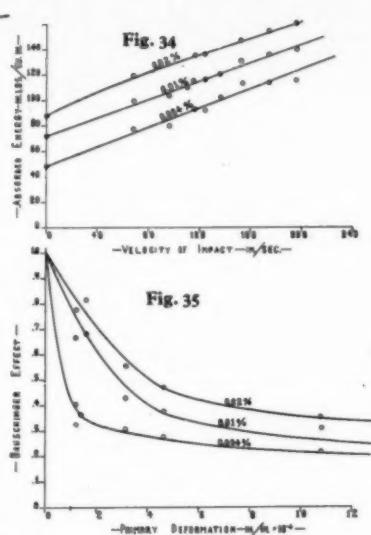


Fig. 34

Fig. 35

and about 50,000 lb./sq. in. are necessary to obtain a permanent deformation of 0.0001% (Fig. 30). The shear modulus of elasticity for Monel metal was found to be about 10,700,000 lb./sq. in. Also, the energy absorbed by Monel metal to produce a small permanent deformation is higher than for nickel-chrome steel, for instance. According to Fig. 31, about 20–25 lb./sq. in. are necessary to produce a permanent deformation of 0.0001% , and about 70–72 lb./sq. in. for a deformation of 0.0003% . For a 0.0002% deformation we applied more than twice the load than is necessary to produce the same deformation in nickel-chrome steel.

In Figs. 32 and 33 are reproduced two characteristic dynamic loading diagrams. Fig. 32 shows a constant speed due to a given dropping height with a varying weight of the hammer. The results of a higher speed produced by a dropping height of 50 in. is represented in Fig. 33. The reversed dynamic torsional test results of the same specimens are represented in the curves No. 2 in these two Figs. For this material, the dynamic torsion loads are fairly high so as to provoke small permanent deformations. About 60 in.-lb./cu. in., under a dropping height of 20 in., produce a permanent deformation of 0.00001% ; and about 120 in.-lb./cu. in. produce a deformation of 0.0001% (Fig. 32). After a total permanent deformation of 0.00151% , the specimen also showed, under small dynamic loads produced in the opposite sense, rapidly increasing

permanent deformations (20 in.-lb./cu. in. for 0.0001% , and 41 in.-lb./cu. in. for about 0.0003%). For higher speeds (50 in. dropping height) the results are shown in Fig. 33. This diagram makes it clear that higher energy is required under higher deformation speed to attain the dynamic elastic limit of the material.

The influence of the speed (varying between 70 in./sec. and about 200 in./sec.) on the necessary energy to produce 0.004% , 0.01% and 0.02% permanent deformation of Monel metal, is shown in Fig. 34. Contrarily to results with nickel-chrome steel, the speed effect is fairly high, and the dynamic elastic limit of this material increases rapidly under higher speeds. For instance, under a loading speed of about 70 in./sec. a dynamic load of about 75 in.-lb./cu. in. is necessary, while a much higher elastic load of about 125 in.-lb./cu. in. can be applied at a speed of 200 in./sec. to attain the dynamic elastic limit with a permanent deformation of 0.004% . Similar results have been recorded for a greater permanent deformation, that is, 0.01% and 0.02% showing also, between the lower and higher speed, an increase of about the same order (40 in.-lb./cu. in.). These curves show also that there is a close agreement between the results of the elastic limit at zero speed and the dynamic test results.

The effect of Bauschinger for this material is shown in Fig. 35, and has also been recorded for three different permanent deformations, of 0.004% , 0.01% and 0.02% . Here it also

becomes clear that, under reversed stresses after a preliminary overloading of the specimen, very low dynamic loads cause an important permanent deformation. A preliminary deformation of 0.003% results in a loss of about 50% of the dynamic load to attain a permanent deformation of 0.01% , and only 25% of the initial dynamic load is needed if the specimen has been permanently deformed to an amount of about 0.0011% (Fig. 35). If the elastic limit is measured under a permanent deformation of 0.004% even smaller dynamic loads are necessary to produce a permanent deformation of the specimen.

In Table II some characteristic numerical results of static and dynamic tests are shown to compare the elastic limit and the effect of Bauschinger of the tested materials.

A comparison of the effect of Bauschinger on the dynamic elastic limit of the five tested materials is given in Fig. 36, for a permanent deformation of 0.01% . According to these diagrams, duralumin 24ST seems to be in a large measure more resistant to preliminary permanent deformation in an opposite sense than any one of the other four materials. For instance, with preliminary permanent deformation of 0.0005% , in one sense, the locked-up stresses in mild steel and in nickel-chrome steel are so high that, under a load in opposite sense of about 8%, respectively, 20% of that necessary to cause a permanent deformation of the same amount in the virgin materials, the elastic limit (0.01%

TABLE II.

Material tested	Static Test			Dynamic test					
	Elastic limit lb./in. ²	Energy in. lb./cu. in. at perm. def.		Modulus of elasticity G. lb./in. ²	Elastic limit at 0.01% perm. def. ft. lb./in. ²		Effect of Bauschinger in % at 0.01% perm. def. Preliminary deformation in %		
		0.01%	0.1%		10 in./sec.	50 in./sec.	0.00025	0.0005	0.001
Duralumin 24 ST.	17,000-17,500	25	43	4,000,000	26	40	22	33	45
Magnesium Alloy 57S . . .	3,600-5,300	0.7-2.8	1.8-3.4	2,800,000	2.4	5.0	45	73	84
Steel, Mild, 1020	25,000	16.5	18.5	10,750,000	50 in./sec. 31	100 in./sec. 39	81	92	95
Ni-Cr Steel, Annealed . . .	33,000-34,000	30	65	11,300,000	50 in./sec. 66	100 in./sec. 72	64	81	91
Monel Metal K, as drawn .	50,000	20	72	9,500,000	50 in./sec. 90	200 in./sec. 142	51	66	74

deformation) is attained. For magnesium alloy 57S, about 27% is needed, for Monel metal 35%, and for duralumin the astonishing high amount of 68% is required, to produce the same effect. These results are important and must certainly be taken into consideration, not only when the material is loaded under reversed dynamic loads, but especially when repeated loads are applied during longer periods, as this takes place, for example, in most parts of machines working under fatigue loads.

This fundamental question will be taken into consideration later on, when repeated impact fatigue tests will be carried out with these materials on the newly devised and constructed impact fatigue machine, built especially for studying research problems of this kind.

Résumé

Because of the fact that a great variety of structural materials is used to-day in high-speed machines, it becomes more and more necessary to determine the characteristics of these materials under very short duration of the applied load. Torsion is recognised as a frequently encountered type of loading in modern machinery. A new testing technique has been developed permitting investigation of the static properties of these materials and especially of their behaviour under dynamically applied loads. To study

specimens, having undergone small permanent deformation by a previous loading in one sense followed by increasing dynamic loads in the opposite sense, a special test set-up was developed. With this apparatus the well-known "effect of Bauschinger," showing that the proportional limit of metallic materials can be changed in wide limits, could be investigated. The energy loads were applied by using either a constant mass and an increasing speed or a constant speed and an increasing mass till the elastic limit was reached. The influence of plastic deformations in one sense on the variation of the elastic limit in the opposite sense has been investigated. Five different alloys, after having measured their static properties in torsion—such as the elastic limit, the energy absorbed, and the shear modulus of elasticity G—were submitted to these special torsion tests and their behaviour under dynamic plus and minus torsion loads was investigated. Dynamic test results show a great variation in the elastic limit of the different materials. Especially the effect of Bauschinger is, for most alloys (as mild steel, nickel-chrome steel, magnesium alloy) surprisingly great, being two to six times more sensitive than for duralumin for instance showing only a low reduction of their dynamic elastic limit under reversed torsion stresses.

It seems that it is of importance to study the behaviour of structural materials under dynamic loads of this kind if the phenomenon of fatigue fracture of machine members, working under repeated dynamic loads, is to be thoroughly understood.

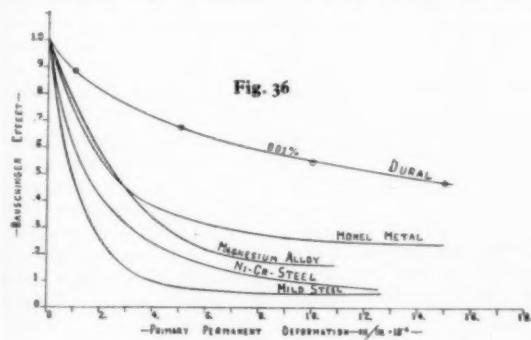


Fig. 36

Changes in Internal Damping of Gas-Turbine Material Due to Continuous Vibration

By J. B. WILKES, JR.

TESTS on four gas-turbine-bucket materials have been conducted at elevated temperatures which involved the measurement of the pressure required to vibrate specimens in a pneumatically-driven fatigue machine. The pressure is assumed to be a qualitative measure of the internal damping of the material.

It was found that at a peak stress of 40,000 psi, nickel and cobalt-base alloys showed a minimum damping near 800° F. and a sharp rise between 1,350° F. and 1,500° F. Two iron-base alloys had a maximum damping between 800° F. and 1,200° F., a slight dip at 1,350° F., and a sharp rise at 1,500° F.

Two of the materials indicated excessively high damping at 1,200° F. and above at stresses below their fatigue limit. This high value, however, became rapidly smaller as vibration was continued to the extent that they finally showed the lowest values of the four materials. This means that damping data obtained by more orthodox means in short times may, in some cases, be very greatly in error if used in choosing materials for gas-turbine buckets. It appears that this could be a rather important factor in those designs where internal damping of the buckets is believed to be a major factor in limiting vibration.

These results are based on a minimum of data and are therefore subject to some revision as more data are accumulated. Probably the largest single source of error is that fact that a number of successive tests were made on a single bar and the results themselves indicate that past history may have a marked effect on behaviour.

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Digest from *Mech. Eng.*, Jan., 1949, 38-39.

The Use of Inhibitors for Controlling Metal Corrosion

Part V—Inhibitors for Light Metals

By G. T. Colegate, B.Sc., A.I.M.

In this concluding article of the series, the author deals with inhibitors for use with aluminium, which is susceptible to attack by alkali as well as by acid, and magnesium. Certain of the inhibitors which have been successfully used with steel are of value in inhibiting the corrosion of aluminium but care is necessary in selecting the correct inhibitors for the corroding medium concerned.

ALUMINIUM, in spite of its position in the electropotential series has, in general, good corrosion resistance and finds many applications on that account. Its resistance to corrosion, both in atmospheric and aqueous media, is due largely to the fact that it easily forms a film of oxide on its surface which is highly protective and which, in the event of it being damaged, has the property of self-healing. Nevertheless, aluminium and its alloys are by no means immune from corrosion and in a variety of solutions it is necessary to use an inhibitor to prevent or minimise the attack. Unlike steel, aluminium is attacked vigorously by alkaline solutions as well as acid ones. Many of the inhibitors that have already been discussed in connection with ferrous metals are also suitable for aluminium.

Corrosion in acid solutions

Aluminium plant is widely used in the chemical and allied industries for handling a variety of acids, mainly organic ones. For instance, acetic acid can be very satisfactorily handled in aluminium equipment, and so can concentrated nitric acid. In all such cases no inhibitor is used. However, if dilute nitric acid is in contact with aluminium, appreciable attack may occur. Eldredge and Mears¹ have shown that small concentrations of chromate are effective in reducing considerably the amount of attack by the acid. The amount of chromate required naturally depends on the concentration of the acid but, in 2%, 5% and 10% acid, 0.1% of chromate will give 98-99% protection, though in 20% acid 0.5% of chromate is necessary.

Sulphuric Acid. Chromates cannot be used to inhibit the attack of sulphuric acid on aluminium. Benson and Mears² found that the presence of

chromate actually increased the attack of the acid considerably. In the presence of chlorides, the stimulation of attack by chromates is greater still. Organic inhibitors have been tried out for use with sulphuric acid but the majority of them are not very effective. There is in fact no inhibitor so far reported which will effectively prevent or even considerably reduce the attack of sulphuric acid on aluminium and its alloys.

Thus in experiments on a commercially pure aluminium and two aluminium-magnesium alloys, containing respectively 2.5% and 5.2% magnesium, it was found that the loss in weight in all cases was two to three times as great after the addition of 1% sodium dichromate to the 10% sulphuric acid solution. While the addition of 1% sodium chloride to the sulphuric acid had no effect on the corrosion rate, when 1% sodium dichromate was added to this mixture the increase in the rate of attack was twenty or thirty times.

Hydrochloric Acid. In this case too, chromates are not satisfactory as inhibitors. On the other hand, several organic inhibitors are known which give excellent protection, the actual degree of protection, however, varying with the concentration of the acid. Among the most effective, in relatively dilute acid (5%) are several of the amines. Geller³ reports that at 1% concentration methyl, ethyl, propyl or butyl amines and several others, gave over 90% protection in acid of this strength.

However, the protection provided by amines in hydrochloric acid is only temporary and it falls off as the temperature increases. It is claimed that the longer the carbon chain of the amine, the more effective are its inhibiting properties. Jenckel and Woltmann⁴ have shown that in weak

acids beta-naphthoquinoline, acridine, 9-phenyl-acridine and several other substances give a very high degree of protection. Wetting agents have also been found to give good protection under some conditions.

Phosphoric Acid. The position in this case is rather different from that with hydrochloric and sulphuric acids, in that chromates are effective in giving good protection over a wide range of concentrations. The amount required varies with the concentration but, in general, if the acid strength is below about 20%, then 0.5% of sodium chromate will give a high degree of protection. Above this strength, however, double the quantity of chromate should be used. Several organic inhibitors have been investigated for use with phosphoric acid and many of them have been found effective, though they are, in general, less effective and more expensive than the chromates.

Corrosion in alkaline solutions

Aluminium and its alloys are rapidly attacked by alkaline solutions and as it is frequently necessary to use such solutions in contact with these materials the choice of inhibitors is important. An example of the wide use of alkaline solutions in contact with aluminium is found in cleaning compounds, both domestic and industrial. These are invariably alkaline and without the use of a suitable inhibitor would lead to considerable attack on the metal.

Caustic alkalis themselves are so vigorous in their attack on aluminium that it has not yet been possible to devise a good inhibitor. Chromates have a certain amount of effect, but the concentration required is so high as to rule them out as a practical proposition in most cases. Various organic inhibitors, notably glue, glucose, etc., can be used, but these

1 *Ind. Eng. Chem.*, 1945, **37**(8), 736.
2 *Chem. Met. Eng.*, 1942, **40**, 88.

3 *Z. Metallkunde*, 1936, **28**, 354.
4 *Z. Anorg. Allgem. Chem.*, 1937, p. 239.

5 *Korr. u. Metallschutz*, 1929, **5**, 41.

have to be present in considerable concentration. However, Rohrig⁶ states that agar-agar and gum arabic in a 0.75% concentration are both sufficiently effective to give more than 80% protection over a fairly wide range of concentration.

Lichtenberg⁷ claims that certain dyestuffs are effective inhibitors in alkaline solutions and Rhodes and Berner⁷ showed that in a molar solution of caustic soda a molar concentration of glucose gave a very large degree of protection, in fact almost completely overcame the attack.

Whereas the inhibition of attack by caustic alkalis is difficult, it is possible in the case of the carbonates, which are widely used in cleaning compounds, to get good inhibition by the use of silicates. Another class of compound frequently used in alkaline cleaners is phosphate. Silicates are also effective in inhibiting attack by phosphates on aluminium. Silicates should not be used in caustic alkali solutions as there is evidence that such additions may increase the attack rather than diminish it. The silicate normally used as an inhibitor is ordinary sodium silicate, but others, such as the metasilicate, have also been shown to be effective, especially for certain purposes. The concentration of silicate required to inhibit attack in both phosphate and carbonate solutions depends on the concentration of the active ingredient and also, very markedly, on the temperature, higher temperatures necessitating the use of a greatly increased amount compared with the lower ones. In solutions just below boiling point the amount of silicate requires to be between a third and a half of the concentration of the carbonate or phosphate.

Chromates are also useful inhibitors in carbonate and phosphate solutions, though they are less effective than silicates and more expensive.

Attack by aqueous solutions

In ordinary domestic or industrial waters chromates are very effective inhibitors for aluminium and have been used for many years. As already pointed out in connection with their use in inhibiting the attack on steel, chromates are classed as dangerous inhibitors, and if used in too low a concentration they may accelerate rather than reduce attack and may lead to pitting.

When using chromate as inhibitors

for aluminium a concentration of about 0.1% is adequate in most cases, i.e., sufficient to maintain the medium permanently coloured. This amount needs to be increased ten-fold in the presence of salts of the heavy metals. The presence of such salts has been shown to cause very considerable increase in the attack on the aluminium. Copper is particularly deleterious in this respect, but adequate dosage of chromate will inhibit the attack of copper-bearing water on aluminium.

Nitrates have also been shown to have good inhibiting properties in neutral waters.

A further class of substance which is effective with waters is soluble oil. Kempf and Daugherty⁸ claim that the use of soluble oil in the engine block of motor cars reduces the attack of cooling water on the aluminium cylinder.

An important and well-known use of chromates as inhibitors of the attack on aluminium alloys by aqueous media is in petrol tanks of aircraft. These are designed in such a way that any water which enters them runs into a sump in which is fitted a capsule containing chromates. These are chosen to be of the correct solubility since obviously if they were too soluble the life of the capsule would be inconveniently short, whereas if they were relatively insoluble, there would be difficulty in maintaining an adequate concentration of chromate ion in the water.

A number of colloids have been shown to be effective in reducing the attack of neutral solutions on aluminium and its alloys. Agar-agar and gum arabic have already been mentioned in connection with attack by caustic alkalis, for which purposes they were seen to be at least as effective as any of the other substances which have been investigated so far. Mears and Eldredge⁹ showed that these two substances, and in addition certain others, such as gelatine, were also effective with domestic waters and dilute salt solutions, though there is always a risk that attack will be intensified rather than reduced. They did their experiments on three materials, namely 99.0-99.4 purity aluminium, an aluminium-manganese alloy containing 1.2% of manganese and on an alloy of the duralumin type containing 4% of copper, 0.5% manganese and 0.5% magnesium.

Other workers have found that agar-agar is an effective inhibitor of the attack of tap water on aluminium. For example, Tinkler and Masters¹⁰ showed that it reduced the attack of hot tap water to one third of what it was in the absence of agar-agar. Friend and Vallance¹¹ examined the effect of additions of gelatine to 3% sodium chloride solutions and found that 0.1% of it was sufficient to reduce the attack by more than a half.

There is evidence that a number of organic compounds of the types already mentioned in connection with attack by acids are also effective in waters. Aronson and Berman¹² investigated the attack on aluminium by distilled water and by 10% potassium chloride solution. In one series of experiments they removed the naturally formed oxide film by abrading it with sand under the surface of the liquids in question and in the other the oxide film was left intact. In the former case it was found that naphthoquinoline and thiocarbamide were effective inhibitors and in the latter case that the use of acridine bases gave good results.

In inhibiting the attack of brines on aluminium, the Aluminium Research Laboratories of America recommend a concentration of sodium dichromate of 1% of the amount of chloride ion present, this amounting in the case of concentrated brines to the same concentration as mentioned for use with steel. The brine should be neutral after the addition of the dichromate and any necessary additions of either caustic soda or hydrochloric acid should be made to bring this about.

Chromates cannot be used with brines if the temperature is high, for example approaching boiling point. Silicates are also effective as inhibitors in brine solutions.

Harris¹³ found that sodium hexametaphosphate is also effective in inhibiting the attack on aluminium of waters containing salts of heavy metals.

Mears and Eldredge⁹ quote several cases from practice on the use of corrosion inhibitors for aluminium. In one instance calcium chloride brine was passed through cooling coils made of aluminium for a period of six years, potassium dichromate being used as an inhibitor. After that period, there was no evidence of attack. In another case, a mixture of ethyl alcohol and

6 *Aluminium*, 1937, **19**, 504.

7 *Ind. Eng. Chem.*, 1933, **25**, 1336.

8 *Automotive Industry*, 1939, **51**, 156.

9 *Trans. Electrochem. Soc.*, 1943, **83**, 403.

10 *Analyst*, 1924, **49**, 30.

11 *J. Chem. Soc.*, 1922, **121**, 466.

12 *Korroziya y Borba s'Nei*, 1940, **6**, 5.

13 *Aluminium and Magnesium*, 1945, **1**, 28.

water was being used as a coolant and was found to cause appreciable attack on aluminium equipment. However, addition of $\frac{1}{2}$ oz./gallon of potassium dichromate to the coolant, almost completely eliminated the attack even when the metal was exposed for extended periods to the liquid at boiling point. Such reduction of the chromate as took place as a result of the action of the alcohol was not sufficient to remove the dichromate completely and did not interfere with its effectiveness. After two years use it was found that the chromate had not only prevented fresh attack but had stopped the attack which had previously occurred.

In the cosmetics and pharmaceutical industries where aluminium containers are used it is necessary to inhibit the attack of any alkaline materials that may come into contact with it. Toothpaste for example, packed in aluminium tubes always contains silicates to prevent serious attack. Silicates are also used in shaving creams. Certain soaps attack aluminium and may be inhibited with silicate in the same way.

MAGNESIUM

Magnesium is a particularly active metal, more so than aluminium, and unlike aluminium it does not form, in the atmosphere or in aqueous media a protective film, so that prevention of corrosion is a very important problem in connection with its use. A great deal of attention has been directed to this matter in the past few years, stimulated by the increasing use of magnesium alloy in aircraft construction and elsewhere. Several inhibitors are found to be effective in preventing the attack of aqueous solutions on magnesium and the use of many of them is covered by patents.

As with aluminium, chromates have been shown to be very effective inhibitors for use with magnesium also. A few tenths of 1% of an alkali chromate or dichromate added to an aqueous, non-reducing solution is sufficient to prevent attack on any of the usual magnesium alloys. Not only is attack prevented on the magnesium alloy itself, but in cases where it is in contact with other, more noble metals, such as copper, the galvanic action between the two is also prevented. It is standard practice to add small quantities of chromate or dichromate to cooling systems which are composed entirely or partially of magnesium alloys and the results obtained are excellent. Chromates may also be

added to solutions used in industry for degreasing and cleaning magnesium alloys. In the form of capsules, chromates are used in petrol tanks and similar containers made of magnesium alloys. In the presence of chlorides, a concentration of chromate should be used corresponding to 10% of the chloride concentration.

Besides the chromates, fluorides have been shown to give good results in protecting magnesium alloy parts from corrosion. In these cases, not only are they effective in preventing attack by aqueous media but also by fuels, especially those containing alcohols. It is also claimed that the use of fluorides is particularly desirable with fuels containing lead tetraethyl as an anti-knock agent. In this case the inhibitor is placed in the lowest part of the tank in the form of a capsule. In aqueous solutions a concentration of 0.5% of alkali fluoride is adequate to prevent corrosion.

Alkali sulphides are another class of substance that has been shown to give good results in preventing, or minimising attack of aqueous solutions on magnesium alloys. The concentration normally used in this case is greater than that for fluorides, being of the order of 1%, though good results are obtained in many cases with lower concentrations than this.

British Patent 381,088 covers the addition of alkali metal sulphides to water, alcohols or non-oxidising saline melts. The patent claims, particularly in the case of alcohols, that the sulphide

addition is effective in preventing corrosion at relatively high temperatures.

The patent also covers the use of sulphides in salt baths used for the heat treatment of magnesium alloys. In this case a concentration of up to 3% of the weight of the salt present is used.

The effectiveness of small additions of sulphides to glycol, glycerine and mixtures of these two substances with water is indicated by the loss in weight figures given in the patent specification. For example, the loss in weight in grams per sq. metre of magnesium exposed to glycol at 100° C. is 19.6 whereas in the presence of 0.1% of potassium sulphide the attack is nil. With glycerine at 150° C. the attack amounted to 9.6 grams per sq. metre, but after the addition of 0.2% liver of sulphur, this was reduced to 0.02 grams per sq. metre.

Chromates are frequently used in paint-type compounds for application to magnesium. Besides paint as such, which, for use with magnesium alloys invariably contains zinc chromate, either alone or in admixture with other chromates, various jointing compounds are used in the construction of equipment and structures of magnesium alloys. These are particularly important for example in aircraft construction where the magnesium is frequently in contact with a dissimilar metal such as aluminium under corrosive conditions. A paint type coating should also be used when magnesium alloys are in contact with wood, etc.

Measurement of Permeability Characteristics of Anodic Films on Aluminium

THESE measurements have been performed on sealed and unsealed anodic films prepared by complete anodising of 99.5% pure aluminium foil in chromic and sulphuric acids and by solution of Al by $HgCl_2$ after anodising. Four different techniques of measurement used concerned the rate of diffusion of salts through the anodic films, the rate of osmosis with the films as a membrane, the rate of transport of water through the film, and the conductance of the films. Results relative to comparable sections of the same sheet agree within 20%; those relative to sections of different sheets diverge by a factor of 2. Sections isolated by $HgCl_2$ gave poor reproducibility; the reproducibility

was, however, very much improved by sealing the film *in situ*. The behaviour of films produced by complete anodising in sulphuric and chromic acids with respect to KCl and KNO_3 is very similar. The "sealing" decreases the permeability by a factor of 100. Anodic films are permeable to solute mols.; mols. as large as sucrose can diffuse through the film. The rate of diffusion of KCl and KNO_3 is found to be equal and proportional to the concentration gradient in the range of 0.1-1.0 M, although their corrosive action is widely different; this proves that the corrosive action of the halide ion is not a result of any special case of penetration of the bulk anodic film. The various results obtained are inconsistent with a structure of parallel open tubes.

From Pittsburgh International Conference on Surface Reactions, pp. 10-20, 1948. (Chemical Abstracts, 42, No. 19, p. 7,222, Oct. 10th, 1948).

Reviews of Current Literature

Technologie Der Leichtmetalle

By Alfred von Zeerleider. Pp. xii + 364, with 62 tables and 396 illustrations. 1947. Rascher Verlag; Zurich. (Available in the German edition only.)

Professor von Zeerleider's new book, although similar to his previous work on the technology of aluminium and its light alloys has been completely re-written, and extended to include magnesium and magnesium base alloys, which are considered at the end of appropriate chapters.

When considering this book, it must be remembered that as in the case of his earlier works, Professor Zeerleider's approach has been an essentially practical one. This is not a metallurgical text book in the sense that readers might hope to find a complete treatise on the constitution and properties of the aluminium and magnesium alloys, but is principally directed to describing the manufacturing processes involved in the production of light alloy components. The chapters cover History and Extraction, Alloys, Properties and Methods of Testing, Design, Melting, Casting, Rolling, Extrusion, Forging, Drawing, Wire Production, Hollowware manufacture, Heat-treatment, Machining, Welding, Riveting, Surface Treatment and a final chapter gives a brief consideration of applications, choice of materials and storage. In addition, there is a classified bibliography, which is additive to and brings up to date that contained in previous editions.

The book contains numerous illustrations, the majority of which are new and improved by comparison with the earlier editions. The tables have been reduced in number but considerably improved in value.

In reviewing a book by an author of the standing of Zeerleider, it is impossible to forget the degree to which the earlier editions have become established and this makes it more difficult to comment objectively on the contents of the new book since certain features have become identified with Zeerleider's books. Nevertheless an attempt will be made to draw attention to the new features to be found in this edition.

Readers familiar with earlier editions will note an alteration in form in that certain subjects which formerly were associated by a common metallurgical denominator have been separated and allocated individual chapters. A particularly gratifying example of this is to be found in Chapter 13, which is devoted to a consideration of heat treatment. A minor re-arrangement of the chapters will also be noted, and this, together with a division into sections containing chapters on related subjects, provides an improved measure of co-ordination.

The book presents essentially a practical treatment of the manufacturing processes involved in the production and working of aluminium and magnesium alloys. The theoretical considerations underlying these processes are only briefly discussed, leaving the reader with the desire to ask for more. It would considerably enhance the value of this book if Professor Zeerleider had devoted more space to a metallurgical study of these materials, of which he has an unrivalled knowledge and experience. It might be suggested that, excellent as many of the illustrations are, some could have been omitted thus permitting more space to be devoted to theoretical matters.

Reference has already been made to the separate chapter on heat treatment, which seems to place this

exceedingly important aspect in a more realistic relation to the other processes than is the case with earlier editions. The heat treatment of aluminium alloys is an extremely precise and important operation, calling for specialised furnaces and temperature control equipment. The new chapter presents a fairly complete survey of the types of furnaces which are used in light alloy manufacture but one wonders if a little more space might have been devoted to temperature measuring and controlling equipment now available and to a more detailed consideration of the theory of heat treatment, principally to emphasise more fully the need for the specialised equipment discussed. Readers would probably be prepared to accept a sacrifice of some illustrations if the text were correspondingly lengthened.

The chapter on casting has been considerably improved by comparison with earlier editions and the illustrations have been extremely well chosen. In view of the present universal usage of the continuous method of ingot casting one might have expected more detail and illustration of this process, particularly the semi-continuous method which has been very widely adopted. This is a small point however, as the process and its modifications are briefly discussed and to many readers the details are probably sufficient.

Discussion of magnesium base alloys constitutes a new feature in this edition and in some respects the introduction of these materials is to be regretted. There has not been a substantial increase in the general text, thus suggesting that by introducing magnesium-base alloys other matter has suffered. "Zeerleider" has always been regarded as an "aluminium" book and in this capacity has attracted a well-deserved reputation. It can be argued that extending the scope of the book, without effective enlargement, to include the magnesium light alloys may injure this reputation and create a situation which the reviewer would greatly regret.

In attempting to present a factual review of the new "Zeerleider" criticism of certain features has been expressed and it must be stated in conclusion, that the merits of Professor Zeerleider's published contributions to the literature of the aluminium alloys, being so well known to many readers, need no additional praise beyond stating that his previous standards are fully maintained in the present book.

C.W.

A Metallurgical Study of German Aircraft Engine and Airframe Parts

Vol. II

By the Aero Component Sub-Committee of the Technical Advisory Committee of the Special and Alloy Steel Committee. Manchester, 1948. The Kennedy Press, Ltd. x + 110 pp., 128 figures. Price 10s. 6d., post free.

THIS volume concludes a very complete summary of data resulting from the metallurgical examination of German aircraft engine and airframe parts by an Aero Components Sub-Committee appointed by the Technical Advisory Committee of the Special and Alloy Steel Committee for the purpose. The first volume, published in 1943, contained data from an extensive range of parts examined from various types of German aircraft. But, at that time, investigations were still in progress and, indeed, were continued to the end of hostilities and to present the conclusions of the Committee this second volume fulfils a very useful function.

Those familiar with the first volume will remember that the object of the investigations was to obtain data on the types and quality of materials used, methods of manufacture, efficiency of the heat treatment to which the parts had been submitted, together with any other information which might prove of value, as, for example, details of finish. But an important aspect of the investigations was to discover the influence of restrictions, due to the blockade, on German procedure and selection of materials; the effect of which is more clearly shown by comparison with this second volume.

As in the first volume, attention is directed chiefly to engine parts, but a number of airframe and miscellaneous parts are included. Special features concerning design were noted in some instances, but the primary objects of the investigations were possible changes in the composition of materials and their effects on the service of the aircraft. Components of the same type from different aircraft have been considered together, and the main features summarised from the individual reports. The manner in which the great amount of detailed information, obtained from these investigations, has been condensed, without sacrifice of the objective, reflects great credit on the Committee. It is probable that few readers of the present volume will appreciate the amount of work involved and how meticulously it was carried out.

The outstanding features of the examination of crankshafts were changes of composition due to shortages of nickel and molybdenum. Instead of using the well-

known 2% nickel, 2% chromium steel, all crankshafts of the Junkers Jumo had been made in 2½% chromium nitriding steel and molybdenum had been replaced by vanadium, with the result that noticeable embrittlement had occurred during nitriding. In the case of crankshafts of Mercedes Benz and Daimler Benz the same change to 2½% chromium steel was made and also a change over to nitriding from carburising. A slight deterioration in quality of steel was noted, but no steels of any unusual composition had been employed.

The conclusions reached from the examination of other parts reported appear to be consistent with a steadily worsening alloy supply position or with a desire to economise in time spent on fabrication. It is noteworthy, however, that, in the case of the cylinders and cylinder liners, samples showed the maintenance of the high standard of quality and workmanship described in the first volume.

Apart from the information on steels used in German aircraft this volume, together with the initial volume, provides an excellent example of the presentation of reports of this type. They are very complete and are both lucid and informative and could be profitably used as a model on which to base reports of similar investigations. Those who have a copy of the first volume should not fail to obtain a copy of the present volume. It is noted that profits from its sale are to be credited to the Royal Air Force Benevolent Fund.

W. A.

Iron and Steel Board

As was announced in our October, 1948, issue the members of the Iron and Steel Board, with the exception of the T.U. members, were unable to accept the invitation of the Minister of Supply to serve a further year, in the changed circumstances likely in their view to arise from the Government's proposals for bringing sections of the iron and steel industry under public ownership. It was, however, also announced that the Board would continue in office pending completion of their work on certain outstanding major problems then in hand. A stage has been reached when the Board feels that this work has now been completed as far as practicable, and accordingly the appointment of the Board terminated on March 31st, 1949.

It is now announced that the executive work previously carried out under the Board will be performed by a division of the Ministry of Supply to be known as the Iron and Steel Division. That division will be in the charge of an Under-Secretary and will be staffed by those previously serving the Iron and Steel Board. Matters connected with the Iron and Steel Bill will continue to be dealt with by a separate division of the Ministry.

Scholarship—University of Durham

It is widely recognised that the future well-being of this country depends on the steady influx of technically trained personnel into industry. A Company which has long maintained this fact, and has done much to give practical support to it, is The Power-Gas Corporation, Ltd., of Stockton-on-Tees. This support is shown in the announcement that this Company has endowed an Open Scholarship, tenable at Durham University, to enable suitably qualified students to undertake a course of study, leading to a degree, in either civil or mechanical engineering. The Scholarship will normally be tenable

for a period of three years but, in exceptional cases, it may be extended for a fourth year.

In 1947, The Power-Gas Corporation endowed an Open Scholarship at the University of Leeds for gas, chemical, mechanical or civil engineering. Here, again, the Scholarship was tenable for a period of three years, with a possible extension for a fourth year.

An Aluminium Lined Fish Package

ARISING from a reference in our recent February issue to the use of aluminium fish boxes, an interesting and informative communication has been received from Mr. C. W. Saunders, general manager of Hull Merchants' Amalgamated Box Co., Ltd., which incorporates the Hull Box Pool and issues some five million packages per year in Hull alone, stating that his Company is changing over completely to a new type of package.

This new package is a liner crate, the liner of which is high purity aluminium. For the initial stages of its development the industry was rather afraid of the thin gauge and softness of the liner material and openly stated that, without anodising, the liners would rapidly deteriorate. The fact that the Company is now changing over completely to this package, after eighteen months' constant use of large quantities under normal conditions, is sufficient contradiction of the earlier criticisms.

According to Mr. D. W. Hill, a member of the Council of the National Federation of Fishmongers, Grimsby has lost its place as the premier fishing port for quality and is lagging behind Hull in the type of container used for transporting fish inland. He expressed the opinion that the introduction of the aluminium liner crate is the biggest advance yet made in the transport of fish. With these crates, said Mr. Hill, fishmongers do not get ragged fish, the keeping qualities seem much more pronounced and the psychological effect is considerable.

The Use of Solochrome Cyanine as a Colorimetric Reagent for Beryllium

With Special Reference to the Determination in Magnesium and its Alloys

By C. H. Wood and H. Isherwood

Solochrome cyanine R.200, a dyestuff which was known to give a colour reaction with aluminium, has been found to act in a similar manner with respect to other metals, under suitable conditions. In this article a method of determining the beryllium content of magnesium alloys is presented. Certain metals, if present in the alloy, interfere with the direct determination and details are given of a preliminary separation using tannin.

WHILE investigating colorimetric methods for the determination of traces of impurity metals in pure magnesium and Elektron alloys, it was found that solochrome cyanine R.200, an I.C.I. dyestuff which was known to give a colour reaction with aluminium,¹ also gave colour reactions with other metals in acid solution, though at somewhat different pH values. Among these metals was beryllium and, as the need arose for the determination of this element in small quantities, it was decided to investigate the use of this dye. It was found that aluminium, ferric iron, ferrous iron, thorium, titanium and zirconium interfered with the direct determination of beryllium at pH 5.8 and that, in the presence of any of these elements a preliminary separation was necessary. Zinc, manganese, rare earth metals, alkaline earth metals and magnesium did not interfere.

Properties of the Reagent

Preliminary investigations of the reactions of solochrome cyanine had shown that the metal lakes, which formed at the same pH as that at which the hydroxide of the metal begins to precipitate, usually showed strong absorption bands at wavelengths in the region of 490–560 m μ .

Fig. 1 shows the characteristic absorption bands in the spectra of the aqueous solutions of the metal complexes investigated, all formed at pH values less than 6.0.

In Fig. 2 are shown the characteristic absorption spectrum of the beryllium complex (1), and the reagent (2), at different concentrations (pH 5.8). The first spectrum in each case is water.

In the preparation of the solutions of the metal complexes, a large excess of the metal was used so that no free dye remained. It was found that at the concentration used the dye showed only slight absorption, but at higher concentrations there was considerable absorption up to 540 m μ .

Development of the Method

In the investigations which follow, the instrument constants given with some of the details refer to the Hilger Spekker Photoelectric Absorptiometer which was used throughout the experiments. Ilford Spectrum Green filters No. 604, which transmit in the region of 520 m μ , were used to give maximum sensitivity.

1 F. Alten, W. Weiland, E. Knippenberg, (*Z. anal. Chem.*) (1934), **93**, 91.

T. Münker, (*Z. anal. Chem.*) (1938), **113**, 83.

"Organic Analytical Reagents", Vol. IV., 1948, pp. 355 *et seq.* Frank J. Welcher.

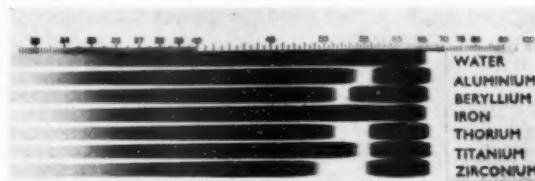


Fig. 1.—Characteristic absorption bands in the spectra of aqueous solutions of the metal complexes.

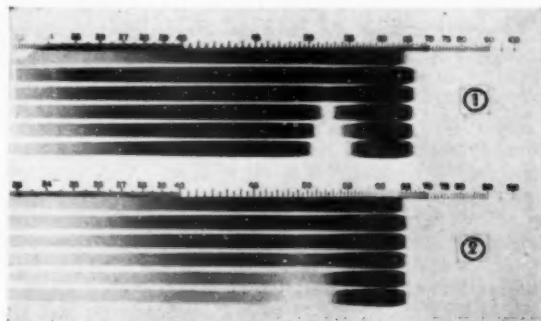


Fig. 2.—Characteristic absorption spectrum of the beryllium complex (1), and the reagent (2), at different concentrations (pH 5.8).

All experiments were carried out in the presence of magnesium as the main reason for the investigation was the application of the method to magnesium and its alloys.

TABLE I.

mg. Beryllium.	Absorption %	Increase in Absorption % for each 0.01 mg. Beryllium.
0.01	20.0	6.0
0.02	26.0	6.0
0.05	32.0	6.0
0.04	38.0	6.0
0.05	43.5	5.5
0.06	48.0	4.5
0.07	52.0	4.0
0.08	56.0	4.0
0.10	62.0	3.0
0.15	71.0	2.0

Instrument Constants : Water/Water 1.00,
Filters : Ilford Spectrum Green No. 604,
Cell 1 cm.

Each test solution contained : 0.1 g. magnesium ; 1.75 ml. of N-sulphuric acid and enough sodium acetate to give a pH of 5.8.

In any colorimetric determination it is necessary to have an excess of the reagent present in the solution, but as the reagent in this case is highly coloured, a large excess is to be avoided if maximum sensitivity is required. Table I gives the percentage absorption obtained when 3 ml. of 0.05% solochrome cyanine solution were added to varying quantities of standard beryllium solution.

It will be seen from the above table that the increase in absorption per 0.01 mg. beryllium falls off for quantities exceeding 0.04 mg., indicating that insufficient reagent was present for this amount of beryllium. It was assumed, therefore, that about 3 ml. of reagent were sufficient for 0.03 mg. beryllium.

As a basis for further work, 10 ml. of the reagent were used for quantities of beryllium up to 0.08 mg.

Having established conditions for satisfactory colour development, a graph was prepared using solutions of varying beryllium content, the details of which are given in Table II:—

TABLE II.

mg. Beryllium.	Drum Difference Reading.
0.00	0.00
0.01	0.11
0.02	0.20
0.03	0.31
0.04	0.41
0.05	0.51
0.06	0.61
0.07	0.72
0.08	0.80

Instrument Constants: Water/Reagent... 1.00.

Filters : Ilford Spectrum Green No. 604.

Cell 1 cm.

Each test contained : 3.5 g. sodium acetate; 0.1 g. magnesium as sulphate; 10 ml. 0.05% solochrome cyanine solution; and 1.75 ml. N/1 sulphuric acid. pH of solution was 5.8.

Separation of Interfering Elements

Since the purest magnesium is seldom entirely free from aluminium and iron, it was necessary to effect a preliminary separation of the beryllium. A study of the literature² revealed that tannin was probably the most suitable reagent since aluminium, iron, titanium, thorium, and zirconium could all be removed from acid-acetate solutions leaving the beryllium in solution. Synthetic mixtures containing elements likely to be met with in magnesium alloys were then made up and the orthodox tannin separation carried out as follows:—

To a solution of 0.2 g. magnesium, as sulphate, contained in a 250 ml. beaker, were added the various amounts of impurity metals listed in Table III. Then 3.5 ml. N/1 sulphuric acid were added followed by 5 ml. 1% tannin solution and enough sodium acetate solution to give a pH of 4.8. The whole was then heated to about 80° C. and kept on the hot-plate for 15 minutes until the precipitated impurities had coagulated. The solution was cooled to room temperature, made up to 100 ml. with distilled water, and filtered through a No. 40 dry-fluted Whatman filter paper. Then 50 ml. of the clear

TABLE III.

Solution No.	Aluminium Present mg.	Zirconium Present mg.	Iron Present mg.	Beryllium Present mg.	Beryllium Found mg.	Error mg.
1	1.00	0.00	0.00	0.000	—	—
2	0.40	0.40	0.40	0.040	0.020	±0.002
3	1.00	0.40	0.00	0.080	0.040	±0.002
4	0.40	0.00	1.00	0.100	0.054	±0.004
5	1.00	0.00	0.40	0.120	0.063	±0.003
6	0.60	1.00	0.40	0.140	0.072	±0.002

² B.D.H. Book of Organic Reagents", 1946. M. L. Nichols, J. M. Schempt, *Ind. Eng. Chem. (Anal. Ed.)*, (1939) 11, 278.

filtrate, equivalent to 0.1 g. magnesium, were transferred to a 250 ml. beaker and treated with enough sodium acetate solution to give a pH of 5.8. Then 10 ml. 0.05% solochrome cyanine solution were added, and the whole diluted to 100 ml.

After standing 15 minutes a reading was taken on the Absorptiometer. The beryllium was calculated from the graph prepared from Table II. The results are shown in column six of Table III.

It will be seen from the above table that the beryllium found is half the amount added; this is because the solution was diluted to 100 ml. for the tannin precipitation, only 50 ml. of which was used for the actual beryllium determination.

Table IV gives the results obtained by dissolving actual samples of pure magnesium and adding various amounts of beryllium to the solutions obtained.

TABLE IV.

Sample No.	Weight g.	N/1 Sulphuric Acid Added.	Beryllium % Present	Beryllium % Found
0	0.206	20.6 ml.	—	—
1	0.197	19.7	0.010	0.010
1a	0.225	22.2	0.010	0.009
2	0.185	18.5	0.022	0.023
2a	0.214	21.4	0.022	0.022
3	0.191	19.1	0.028	0.030
3a	0.203	20.3	0.028	0.027
4	0.187	18.7	0.036	0.040
4a	0.182	18.3	0.036	0.036
5	0.184	18.4	0.049	0.050
5a	0.188	18.8	0.049	0.052
6	0.184	18.4	0.062	0.068
6a	0.201	20.1	0.062	0.065

During these determinations it was found that the pH of 4.8, required initially for the removal of the impurities, was rather critical. At a pH of 4.6 zirconium and iron appear to be satisfactorily removed, but small quantities of aluminium remain in solution and are precipitated on the addition of the second quantity of sodium acetate—i.e., when the pH is raised to 5.8. If the aluminium is filtered off at this stage, the solution can then be used for the determination of beryllium, but this procedure is not recommended.

At a pH of 5.0 and over, all the impurities are removed but slightly low results are sometimes obtained, if the total impurities exceed about 1%. No doubt this is due to co-precipitation of some beryllium.

By following precisely the detailed procedure given below, particularly with regard to the quantities of reagents, such as normal sulphuric acid and sodium acetate, no difficulties have been experienced in obtaining the correct pH values, which were checked on a Cambridge pH Meter using a glass electrode.

Detailed Methods

Reagents :

Sodium acetate, 20% solution.
N/1 sulphuric acid solution.

Solochrome cyanine, 0.05% solution in water.
Tannin ... 1% solution in water (freshly prepared).

Method 1.—About 0.2 g. of sample is transferred to a 250 ml. beaker and dissolved by the addition of exactly 100 ml. normal sulphuric acid per gram of metal taken.

When dissolution is complete, 5 ml. tannin solution are added, followed by 5.0 ml. sodium acetate solution to bring the pH of the solution up to 4.8 when diluted to 90–95 ml. The solution is heated to 80° C. and digested on the edge of the hot-plate for 15 minutes, or until the precipitate has coagulated. The solution is then cooled, and diluted to 100 ml. in a graduated flask.

The precipitate is filtered off on a dry Whatman No. 40 fluted filter paper, and 50 ml. of the filtrate (representing 0.1 g. sample) are transferred to a 100 ml. graduated flask containing 13 ml. sodium acetate solution. Then 10 ml. solochrome cyanine solution are added and the solution diluted to 100 ml. with distilled water. After standing for 15 minutes a portion of the solution, which should have a pH of 5.8, is transferred to a 1 cm. cell and a reading taken on the Spekker Absorptiometer.

A blank is run at the same time as the samples using high purity magnesium. This is used to set the instrument.

INSTRUMENT CONSTANTS.	
Beryllium % ..	0.01-0.07
Filter ..	Ilford Spectrum Green No. 604
Cell ..	1 cm.
Water/Blank Setting ..	1.00
NOTE :—Water/water setting should be about 1.55.	

Method 2.—For quantities of beryllium below 0.01% in pure magnesium a larger sample is taken. This necessitates a preliminary separation of the beryllium since high salt concentrations suppress the colour of the beryllium solochrome cyanine complex and therefore reduce the sensitivity.

2 g. or more of the sample are transferred to a 400 ml. beaker and dissolved by the addition of 20 ml. distilled water and 10 ml. concentrated hydrochloric acid per gram of metal. Then 2 g. ammonium chloride are added, followed by dilute ammonia until the solution is just alkaline to litmus. A thin cream of paper pulp is stirred into the solution, which is heated to 60°-70°C. Then 10 ml. 1% tannin solution are added, followed by 1 ml. of a standard iron solution containing 0.5 g. iron/litre. After standing for 30 minutes at 60°C., the solution is filtered on a No. 40 Whatman filter paper folded inside a No. 541. The precipitate is washed with a 1% solution of ammonium nitrate, made just ammoniacal and then is sucked dry on the pump. The No. 40 filter paper containing the precipitate is transferred to a platinum or silica crucible and ignited at 700°-800°C. The residue is fused with 0.5 g. potassium bisulphate and the cooled melt extracted with a little distilled water containing 1.75 ml. normal sulphuric acid. Then 2.0 g. magnesium sulphate are added (equivalent to 0.2 g. magnesium), followed by 5 ml. tannin solution and 5.0 ml. sodium acetate solution, or sufficient to bring the pH up to 4.8 when diluted to 100 ml.

The solution, diluted to 90-95 ml., is heated to 80°C. and digested on the edge of the hot-plate for 15 minutes, or until the precipitate has coagulated. It is then cooled and diluted to 100 ml. in a graduated flask.

The procedure from this point is the same as in Method 1.

INSTRUMENT CONSTANTS	
Beryllium % ..	0.001-0.01
Filter ..	Ilford Spectrum Green No. 604
Cell ..	1 cm.
Water/Reagent Setting ..	1.00

Notes on the separation of large amounts of Aluminium and Zirconium.—(1) In the presence of large amounts of aluminium (such as Elektron A8, AZ91 alloys) it is necessary to carry out the following procedure:

The solution of about 0.5 g. of the alloy in hydrochloric acid, is treated with ammonium chloride and ammonia in the presence of paper cream to precipitate Group 3 elements, including beryllium. The precipitated hydroxides are filtered off, dried and ignited in a platinum crucible. They are then mixed with 5-10 g. sodium carbonate and fused over a Meker burner for half-an-

hour. The cooled melt is extracted with hot water and filtered. The residue (containing the beryllium) is washed well with hot water and then ignited in a silica or platinum crucible. It is fused with 0.5 g. potassium bisulphate and the extract treated as described in the above method. (2) In the presence of large amounts of zirconium (such as ZW2, ZW3, Z5Z, etc.), the following procedure should be adopted:

The solution of about 6 g. of the alloy in hydrochloric acid is treated with ammonium chloride and ammonia in order to precipitate Group 3 elements and beryllium. The precipitate is filtered off, dissolved in hydrochloric acid, and reprecipitated. The hydroxides are then dissolved in 25 ml. 50% sulphuric acid and diluted to 250 ml. The solution is cooled to 10°C. and an excess of a 6% solution of cupferron added, while stirring vigorously. The precipitate is allowed to settle, filtered off on a pulp pad, and is then washed with normal hydrochloric acid containing a little cupferron. The precipitate is rejected. To the filtrate are added 2 g. ammonium chloride and enough ammonia to render the solution just alkaline to litmus.

From this point the tannin separation of the impurities is carried out as described in Method 2.

The authors' thanks are due to the Directors of Magnesium Elektron, Ltd. for permission to publish this paper.

Obituary—Mr. W. F. Rowden

We regret to announce the death of Mr. W. F. Rowden, a director of the Climax Molybdenum Company of Europe, Ltd. and a well-known authority on the development of alloy steels, the practical aspects of which he had been associated with from his early days in industry.

Born on May 30th, 1897, he began his industrial career in the Newcastle and Openshaw Works of Armstrong Whitworth & Co., Ltd. and remained for several years, ultimately concentrating on steelmaking. Later he joined High Speed Steel Alloys, Ltd., which had previously been formed by British tool steel makers, as technical adviser. In this connection he maintained contact with practically all concerned with the use of many alloying elements used in steelmaking. Early in 1937 he joined the staff of the Development and Research Department of the Mond Nickel Co., Ltd., and at the end of 1938, he was largely responsible for organising the European Company of the Climax Molybdenum Company, becoming a director of the then newly-formed Climax Molybdenum Company of Europe, Ltd. He made frequent journeys to the United States, maintaining a close liaison with American practice, through his parent company. Indeed, it was from the last of these journeys, towards the end of 1948, that he returned a sick man and from which he did not recover.

During the war he rendered valuable service as a metallurgical adviser to the Department of Tank Design, Ministry of Supply. His work during the major part of his career kept him in constant touch with metallurgists and engineers in many industries, particularly steelmakers, iron and steel founders and users of iron and steel castings and of wrought steel, and his quiet likeable personality and the unstinting helpfulness, which he gave from his wide experience, gained him many friends, by whom our regret will be shared.

He was associated with a number of technical institutes and societies.

The Institute of Metals

Annual General Meeting

For some time now there has been a desire for the presentation and discussion of papers with a practical outlook at the meetings of the Institute of Metals. The Capper Pass and W.H.A. Robertson Awards have been made with a view to encouraging the submission of such papers, and the Institute itself took a major step forward in arranging for the holding of a whole-day Symposium on "The Metallurgical Aspects of Non-Ferrous Metal Melting and Casting of Ingots for Working" as part of the Annual General Meeting held at the end of last month. That the venture was appreciated was indicated by the large number of members who attended and no doubt the Institute will be encouraged to proceed with arrangements for further symposia of a similar nature.

THE Annual General Meeting of the Institute of Metals was held in London on March 30th and 31st, and April 1st. In the unfortunate absence of the President, Sir Arthur Smout, due to illness, the Chair at the opening session was taken by Colonel Sir Paul Gueterbock, Past-President, who extended a cordial welcome to overseas visitors to the meeting.

Election of Officers

The Secretary announced the names of members elected to fill vacancies on the Council for the year 1949-50 as follows:—

President : Sir Arthur Smout, J.P. (Director of Imperial Chemical Industries, Ltd.).

Vice-Presidents : Major C. J. P. Ball, D.S.O., M.C. (Chairman and Managing Director, Magnesium Elektron, Ltd.) ; Dr. C. J. Smithells, M.C. (Director of Research, The British Aluminium Co., Ltd.).

Members of Council : Mr. E. A. Bolton, M.Sc. (Factory Manager, Imperial Chemical Industries, Ltd., Birmingham) ; Mr. C. H. Davy (Director of Babcock and Wilcox, Ltd., London) ; Dr. A. G. Quarrell, A.R.C.S., D.I.C. (Research Manager, The British Non-Ferrous Metals Research Association).

The chairman announced that the Council had accepted with regret the resignation, for reasons of ill health, of Professor Leslie Aitchison as an Ordinary Member of Council.

The meeting approved the recommendation of the Council that, in future, the President should serve for a period of one year instead of two, and that the Council should elect a senior Vice-President who would be the Council's nominee for the Presidency the following year. The Council's nomination of Mr. H. S. Tasker, B.A., Chairman of Goodlass Wall and Lead Industries, Ltd., as senior Vice-President for 1949-50 was also approved and Sir Paul Gueterbock thereupon handed over to Mr. Tasker the Chairmanship of the meeting.

Presentation of Institute's Platinum Medal

The Institute of Metals Medal for 1949 was presented to Dr. William Hume-Rothery, F.R.S., in recognition of his outstanding contributions to the science of non-ferrous metallurgy.

Dr. Hume-Rothery was educated at Cheltenham College; the Royal Military College, Woolwich and Magdalen College, Oxford. In 1921 he was awarded a Demyship in Natural Science, and graduated in 1922, with First-class Honours in Chemistry.

From 1922-1925 he studied at the Royal School of Mines, London, under the late Sir Harold Carpenter, and took the London Ph.D. degree with a thesis on intermetallic compounds, in which the first idea of electron compounds was introduced. He was awarded a Senior Demyship at Magdalen College, Oxford in 1925, and carried out research work at Oxford, at first in temporary accommodation provided by the late Professor W. H. Perkin and later in the Inorganic Chemistry Department, first under Professor F. Soddy and later under Sir Cyril Hinshelwood.

Dr. Hume-Rothery was elected to the Armourers' and Brasiers' Research Fellowship of the Royal Society, in 1929. In 1934 he received a Beilby Memorial Award and was elected to a Warren Research Fellowship of the Royal Society, which he has held—except for one year, between two appointments—ever since; in the same year he published, with G. W. Abbott and K. W. Channel-Evans, a paper which led to the idea of atomic size factor. He was elected a Fellow of the Royal Society in 1937, and awarded the Sc.D. degree of Oxford University in the same year. From 1938-1943 he held a Magdalen College, Oxford, Fellowship by special election. Since his election to a Warren Research Fellowship, Dr. Hume-Rothery has published many scientific papers in collaboration with colleagues, students, and research assistants, and notably with Dr. G. V. Raynor and Dr. P. W. Reynolds on the constitution and theories of non-ferrous alloys, in the *Philosophical Transactions* and the *Proceedings of the Royal Society*, the *Journal of the Institute of Metals*, *Journal of the Iron and Steel Institute*, and the *Philosophical Magazine*. During the war he was occupied in extra-mural research for the Ministry of Supply and the Ministry of Aircraft Production, mainly in connection with titanium carbides in steels, electro-deposited chromium, and the constitution of aluminium alloys. In 1946 he was the Annual Lecturer to the American Institute of Mining and Metallurgical Engineers.

He is the author of "The Metallic State" (Oxford University Press, 1931); "The Structure of Metals and Alloys" (Institute of Metals Monograph No. 1); "Atomic Theory for Students of Metallurgy" (Institute of Metals Monograph No. 3); and "Electrons, Atoms, Metals and Alloys" (Ilfie and Sons, Ltd., 1948). He is a Member of Council of the Institution of Metallurgists; and a member of the Metal Physics Committee of the Institute of Metals, the Metal Physics Committee of the British Iron and Steel Research Association, and of the

Metallurgical Advisory Council of the Ministry of Supply and of the associated Basic Properties and Heat-Resistance Committees. He was elected a member of the Institute of Metals in 1923.

In presenting the medal, Mr. Tasker, using the words of the President, said that Dr. Hume-Rothery had not only made brilliant contributions to scientific knowledge, but, what was of equal importance, he had blazed a trail and, like all the great masters, he had trained men to follow in his steps. Several members of the Hume-Rothery School had since distinguished themselves in applying and extending Dr. Hume-Rothery's discoveries.

Dr. Hume-Rothery in reply said he hoped the Chairman would not feel that he was presenting the medal merely to an individual, who was fortunate enough to have his name inscribed on their list. He was presenting it to a large number of other people who had played their part either by direct research, or indirectly by providing a scheme which, when all had been said against it, had enabled research workers in many universities to do the work to which they felt they had been called.

Technical Sessions

Following the conclusion of the business meeting, the remainder of the morning was devoted to discussion of the following papers:—"The Working Behaviour of Phosphorus-Deoxidised Coppers Containing Bismuth," by A. P. C. Hallowes, and "The Damping Capacity of Metals in Transverse Vibration," and "The Effect of Grain Size on the Damping Capacity of Alpha Brass," both by K. M. Entwistle.

In the afternoon the papers for discussion were: "The Flow of Metal in Tube Extrusion," by C. Blazey, L. Broad, W. S. Gummer, and D. B. Thompson; and "The Promotion and Acceleration of Metallic Corrosion by Micro-Organisms," by T. Howard Rogers.

Digest of Papers

THE WORKING BEHAVIOUR OF PHOSPHORUS-DEOXIDISED COPPERS CONTAINING BISMUTH.

By A. P. C. HALLOWES, B.Sc., A.I.M.

IT has been shown previously that oxygen-free coppers containing about 0.002% or more of bismuth are brittle in the range 400°–700° C., and also at room temperature after annealing in this range. The earlier work also indicated that bismuth contents higher than those usually tolerable could be handled without undue difficulty if the temperature of hot working, or of annealing prior to cold working, were substantially increased, and if intermediate annealing were followed by quenching.

In the present paper, the author deals with laboratory and works scale trials on arsenical and non-arsenical coppers containing bismuth. Of the two working schedules adopted for the works trials, one represented the normal practice of the works concerned, whilst the other was a modification in line with the findings of the earlier work. The operations covered included hot piercing of billets, tube drawing and hot rolling.

In the main, the behaviour of the coppers was consistently related to their bismuth contents. Whilst there were minor differences in the behaviour of arsenical and non-arsenical coppers, these were not altogether consistent and it is concluded that for practical purposes they are similarly affected by a given bismuth content.

The whole of Thursday, March 31st, was devoted to a Symposium on Metallurgical Aspects of Non-Ferrous Metal Melting and Casting of Ingots for Working, the papers discussed being: "Melting and Casting of Non-Ferrous Metals," by G. L. Bailey and W. A. Baker; "The Production of Refined Copper Shapes," by R. H. Waddington; "Melting and Casting Aluminium Bronze Ingots for Subsequent Working," by A. J. Murphy and G. T. Callis; "The Application of Flux Degassing to Commercially Cast Phosphor Bronze," by N. I. Bond-Williams; "The Melting and Casting of Brass," by Maurice Cook and N. F. Fletcher; and "The Melting and Casting of Nickel Silver at the Works of Messrs. Henry Wiggin & Co., Ltd.," by E. J. Bradbury and P. G. Turner.

At the closing session on the morning of April 1st, "The Recovery and Recrystallisation of Rolled Aluminium of Commercial Purity," by P. C. Varley was the first paper presented. This was followed by three papers dealing with an investigation into the problems associated with the welding of aluminium alloys: "High Temperature Tensile Properties of Cast Aluminium-Silicon Alloys and their Constitutional Significance"; and "A Consideration of the Nature of Brittleness at Temperatures above the Solidus in Castings and Welds in Aluminium Alloys," both by W. I. Pumphrey and P. H. Jennings, and "A Consideration of the Nature of Brittleness at Temperatures Below the Solidus in Castings and Welds in Aluminium Alloys," by W. I. Pumphrey and D. C. Moore. The final paper discussed was "The Measurement of Grain-Size of Tungsten and Tungsten Carbide Powders used for the Manufacture of Hard Metal," by H. Burden and A. Barker.

In the following notes is presented a digest of the papers themselves and of the discussion they aroused.

and Discussions

Severe hot work should not be performed in the range 400°–700° C. with bismuth contents of 0.002% or more. Relatively mild work is practicable in this temperature range with contents up to and including 0.003%. Temperatures appreciably above the range, e.g., 750°–900° C., are recommended for materials of higher bismuth content, or where the working is severe.

Cold work should be preceded by annealing at relatively high temperatures and quenching, the annealing temperature required varying with the bismuth content and the severity of the working. Severe cold work should be preceded by annealing at, and quenching from, 650° and 750° C. for material containing up to 0.0025% and 0.0025–0.007% Bi., respectively. For mild working, temperatures about 50° C. lower are permissible.

Discussion

The general opinion was held that this was a paper for reference rather than discussion; if the occasion arose when coppers with high bismuth contents had to be used, the detailed information necessary for the avoidance of trouble was available. MR. E. A. BOLTON emphasised the need for manufacturers to pass on to users the data necessary for the fabrication of such coppers, other wise embrittlement could result from faulty treatment by the user. Other speakers referred to the practical difficulties of heat treating at the relatively high temperatures recommended in the paper.

THE DAMPING CAPACITY OF METALS IN TRANSVERSE VIBRATION

By K. M. ENTWISTLE, M.Sc., Ph.D.

THE damping capacity of polycrystalline metals at low stress is independent of both the amplitude of vibration and the number of stress cycles. At any frequency therefore, measurement under these conditions yields results which are uniquely determined by the metallurgical condition of the specimen. In order that such measurements may be of value as a research tool in physical metallurgy, it is essential that a method be developed which gives a reliable indication of the true damping capacity. Details are given of a method for freely suspended specimens in flexural vibration. The most serious source of apparatus loss results from air friction and acoustic radiation and is eliminated by conducting tests at pressures below 10^{-2} mm. Hg.

The damping capacity of aluminium alloys (RR56, RR88 and Duralumin), measured with the fully developed apparatus ranges from 0.0022–0.0035%. These values are somewhat higher than those published by Frommer and Murray for torsional vibration, but the discrepancy can be explained on the basis of Zener's theory which predicts a contribution of 0.0008% from transverse thermal currents in transverse vibration.

The damping capacities of various precipitation-hardening alloys are given for various conditions. The lowest value for RR56 and Duralumin is found immediately after quenching. Room temperature ageing of Duralumin results in a rise to a maximum after 24 hours, and a subsequent fall to a value slightly higher than the as-quenched value. RR56, at room temperature, shows a progressive rise over a period of 14 days, but precipitation at 170°C . for 12 hours produces a decrease in damping capacity similar to that occurring on fully ageing Duralumin. Similar, though less marked effects occur with beryllium-copper alloys.

THE EFFECT OF GRAIN-SIZE ON THE DAMPING CAPACITY OF ALPHA BRASS.

By K. M. ENTWISTLE, M.Sc., Ph.D.

ACCORDING to Zener, thermal currents contribute to the damping capacity of metals. Stress gradients inherent in the form of stressing (other than pure shear) give rise to "macroscopic" thermal currents, whereas the anisotropic properties of metal crystals which, together with even partial random orientation, produce stress differences between adjacent grains, result in intercrystalline heat currents. All contributions resulting from thermal currents are frequency sensitive rising to a maximum value when the periodicity of the vibration approaches the relaxation time for heat flow by conduction between the regions of stress inhomogeneity. The macroscopic effect would be absent in torsional vibration, where the stress is pure shear, but the intercrystalline thermal currents should arise in torsional, transverse and longitudinal vibration. Randall, Rose and Zener have obtained experimental confirmation of the theory in the case of alpha brass in longitudinal vibration, and in this paper the author presents the results of his experiments using torsional and transverse vibration.

As it is the intercrystalline effect which is affected by the grain-size, the specimen size of the transverse tests was chosen with a view to minimising the macroscopic thermal currents. The results provide evidence

of the existence of a contribution to damping capacity in torsional and transverse vibration by intercrystalline currents.

It is concluded that a variation of damping capacity with frequency is to be expected in torsional, longitudinal and flexural vibration. Since it has been shown that the maximum contribution from intercrystalline thermal currents is similar in all types of vibration, then for annealed, single-phase, non-magnetic metals, the principal factor which will cause the low-stress damping capacity to be dependent on the kind of vibration is the macroscopic effect.

Discussion

Several speakers referred to the excellent quality of DR. ENTWISTLE's work on damping capacity and in so doing, DR. HUME-ROTHERY remarked that the accuracy of the work was becoming so high that other factors of the problem would have to be treated with a corresponding degree of accuracy. He agreed with the use of the intercept method for measuring grain size, but felt it was not satisfactory for determining the amount of twinning due to the possibility of an intercept passing through a crystal in such a direction that the twins were not intersected by it. The only satisfactory way was to count all the twins in the circle of view. These remarks provoked a secondary discussion on the measurement of grain size in which MR. W. F. CHUBB advocated the measurement as a volume and MR. B. WALTERS considered the intercept method, due to the rapidity with which it could be carried out, offered possibilities for use statistically.

Turning to the general discussion of the papers, the shift of the emphasis, in damping capacity work, from the engineering aspect to its use as a research tool was referred to by PROFESSOR THOMPSON who mentioned its possibilities in the determination of small variations of elastic modulus. He was most impressed by the complete confirmation of Zener's theory which had been afforded by the work. Referring to the absence of quantitative agreement in ageing curves on two samples, PROFESSOR THOMPSON suggested that incomplete solution treatment might be the explanation. Zener's conception of a relaxation spectrum with a number of peaks at different frequencies corresponding to the effect of various factors contributing to the damping capacity was outlined by MR. M. W. THRING who pointed out that the peak involved in this paper was at the extra fast end of the spectrum. Following up this aspect MR. L. ROTHERHAM referred to the different magnitudes of the peaks due to different forms of relaxation and pointed out that whilst the work of Dr. Entwistle was concerned with one of the small peaks and required very accurate methods, some of them were so large as to be capable of determination with the simplest of apparatus. MR. ROTHERHAM instanced the grain boundary viscosity effect as such a measurement.

The application of damping capacity measurements to a study of precipitation-hardening alloys was referred to by MR. ROTHERHAM who pointed out the need to ensure that the material did not undergo change during the test. DR. HANSTOCK also dealt with this aspect of the work and mentioned that he had obtained similar results to those of Dr. Entwistle, on aluminium alloys, using torsional vibration. He felt that in seeking an explanation of the variations in damping capacity of such materials it would be necessary to undertake further work, varying the frequency and, to a limited extent the temperature, with due regard to the effect of the latter on the material. RR.56 might be a suitable material for the experiments as it was possible

to get quite large increases in damping capacity by room temperature ageing without such subsequent falling off as occurred with duralumin.

DR. HUME-ROTHERY directed the attention of the Publications Committee to the value of a text book on the subject written from a metallurgical point of view and PROFESSOR O'NEILL as Chairman of that Committee stated that such a book was being prepared.

DR. ENTWISTLE, in replying to a point DR. HANSTOCK had raised, referred to the difficulty in obtaining accurate results when the resonance curve is too sharp and the decay time is too short to measure accurately by either method. Measurements involving damping capacities of the order of 0.06% came in this category. On the question of damping capacity from an engineer's point of view, raised by DR. E. VOCE, DR. ENTWISTLE felt that the tests should be carried out by the engineers themselves as the results required would be unique in their application.

THE FLOW OF METAL IN TUBE EXTRUSION.

By C. BLAZHEY, M.Sc., L. BROAD, W. S. GUMMER and D. B. THOMPSON.

FLOW of metal in tube extrusion was studied by means of small pins inserted in the outside of the billets and by means of composite billets. The principal factor determining the type of flow is the degree to which the skin of the billet remains in place against the container wall. In a non-lubricated container, there is sufficient friction between brass and copper-nickel billets and the container wall to hold the skin of the billet in place, but with copper the oxide envelope formed in pre-heating appears to function as a lubricant for the underlying metal. In a lubricated container, brass and copper-nickel flow like copper. When copper is prevented from oxidising it flows like brass in a non-lubricated container, and when a layer of copper is placed around a brass billet the brass flows in a non-lubricated container in the same way as copper.

The presence of a thick oxide layer is not of itself a guarantee that the billet will exhibit the lubricated type of flow; the controlling factor appears to be whether or not the billet can slide within its oxide envelope under pressure in the container.

From a practical point of view, the principal distinction between the two types of flow is that superficial defects on the billet are likely to appear between the walls of the shell with the non-lubricated type and on the outer surface of the shell with the lubricated type, and double or blistered walls are likely to arise with the lubricated type. A sealy inner wall on the shell, due to axial porosity in the billet, is more likely to arise from the use of a pointed mandrel than a blunt or square-ended mandrel.

Discussion

In opening the discussion, MR. C. E. PEARSON referred to the deep seated extrusion defect sometimes associated with the non-lubricated type of flow and the surface defects which may result from the lubricated type of flow. The lubricated type of flow in copper was dependent in some way on the oxidised surface. It appeared from the paper that the scale adhered to the container surface and the copper slid within it as though the oxide-metal interface were lacking in cohesive strength. If one could be confident that the scale would be entirely retained on the container wall the surface defects would also be eliminated. It might also be possible to form suitable oxide films on other copper alloys.

On this question of the properties of oxide films, MR. W. A. BAKER referred to some B.N.F.M.R.A. work on the mechanical properties of scales on coppers at elevated temperatures. According to the type of copper, the scales varied considerably in high temperature ductility; the coppers used by the authors would form a scale which would be plastic under the conditions obtaining at the wall of the container. Due to the high thermal conductivity of the copper and the low conductivity of the scale, the most plastic part of the latter would be at the metal-scale interface which might explain the relative movement of metal and scale at that point. DR. R. GENDERS suggested a further possible explanation of the effect of the oxide. Copper oxide had not the plate-like crystalline structure of most solid lubricants and he considered that the difference in behaviour between copper and brass was due to the fact that the higher conductivity of the former resulted in a hotter skin, more able to flow. In this connection the insulating effect of the copper oxide skin might be quite important. In commenting on this suggestion, MR. PEARSON referred to aluminium alloys which were good conductors, were extruded from heated containers and flowed in a non-lubricated manner.

THE PROMOTION AND ACCELERATION OF METALLIC CORROSION BY MICRO-ORGANISMS.

By T. HOWARD ROGERS, D.I.C., A.I.M.

CORROSION by bacterial action can be brought about in various ways: (a) by direct action, by the production of corrosive substances; (b) by metabolism of the bacteria, producing organic products which can act as depolarisers or catalysts of corrosion reactions; and (c) in cases similar to the anaerobic corrosion of iron, by the action of sulphate reducing organisms, where under some conditions the corrosion reaction is an integral part of the metabolic cycle of the bacteria. In some cases, for instance, where the action results in the production of a catalyst, it is possible for the medium to remain abnormally corrosive even after the bacteria have died or been removed.

In this paper some of the more important aspects of the B.N.F.M.R.A.'s investigation of the influence of bacteria in condenser tube corrosion are discussed. One interesting fact which emerged was that many of the cultures isolated from estuarine waters could withstand concentrations of copper salts in excess of those usually stated to be lethal to micro-organisms.

Considerable attention is paid to the corrosion accelerating action of a mercaptan-disulphide system such as cysteine-cystine, which may result from the bacterial decomposition of organic matter. Cystine and its reduced form cysteine are capable of forming a reversible oxidation-reduction system which might well cause depolarisation at a corrosion centre. In addition films of metallic complexes may be formed on the surfaces of certain metals as a result of the affinity between cystine and the metal; these, too, have an important bearing on the corrosion process.

Sulphate-reducing bacteria have been shown to result in corrosion of non-ferrous metals, as has the presence of slimes which on decomposition give rise to corrosion troubles.

In some cases, inhibition may be effected by non-corrosive dyestuffs, but such methods are only applicable to closed systems. In other cases the problem will have to be tackled by the development of resistant alloys.

Discussion

MR. ROGERS in introducing his paper emphasised the extreme localisation of the production of corrosive substances in the case of a bacterial colony or slime associated with a metal surface and the extremely small amounts of corrosion accelerators necessary for increased attack.

In opening the discussion, DR. U. R. EVANS discussed the action of cystine in the corrosion of copper alloys. Either because of the amount of cystine or due to jet impingement the film of the copper complex formed might cover most but not all the surface and thus leave a small bare anodic area. The cystine-cysteine reaction could then enable the film to act as an oxygen utiliser, and the oxygen to act indirectly as a cathodic stimulator. With the combination of small anode and large cathode severe localised corrosion would result and there were many analogies between these substances and dangerous inhibitors. The production of films which completely covered the metal would reduce the corrosion and addition of zinc salts or the application of a zinc-type paint might give further protection by the deposition of a film of zinc hydroxide on the film.

The question of a standard sea-water was referred to by several speakers. DR. F. A. CHAMPION felt that one of the difficulties in the development of alloys to resist micro-biological attack was the lack of solutions which represented natural conditions in a reproducible way. Both DR. CHAMPION and MR. T. H. TURNER emphasised that there was no *standard* sea water, considerable variations occurring from place to place.

From an engineering point of view, MR. TURNER said, pitting corrosion was more serious than general corrosion and DR. VOCE for copper water pipes, MISS PILS for cooking utensils and MR. BRIMELOW for aluminium water pipes, wondered whether micro-biological corrosion could explain their pitting troubles.

MR. P. T. GILBERT said that much of the work had been carried out in connection with the corrosion of condenser tubes and using the jet impingement test contaminated sea waters showed up as much more strongly corrosive than clean sea water. In addition they had observed quite deep pitting elsewhere than in the jet area. Although in the later stages of the test they got some inhibition in cystine containing sea waters, there was an increase in general corrosion in the early stages and that fact had been used to estimate the corrosiveness of sea waters.

In his reply MR. ROGERS agreed that cystine did act like a dangerous inhibitor but although impingement attack might be inhibited by thicker films, another type of corrosion might result from the instability of the film.

SYMPOSIUM

MELTING AND CASTING OF NON-FERROUS METALS

By G. L. BAILEY, M.Sc., F.I.M. and W. A. BAKER, B.Sc., F.I.M.

THIS paper, reviewing the general principles concerned in the melting and casting of non-ferrous metals and describing the methods commonly used formed an excellent introduction to the series of papers on the subject.

For the production of perfectly sound ingots, attention must be paid to surface quality, internal soundness, structure and internal stress. Folds, splashes, cold shuts, surface inclusions, exudation of low-melting-

point phases and surface cracks are among the surface defects to be avoided in ingots for working. Internal cavities may be the result of shrinkage or gas evolution and, although they may sometimes be eliminated in working, sub-surface defects give rise to "spills" in the finished product. Inclusions may also be harmful, especially where non-metallic phases remain liquid after the metal is solid, as under certain conditions inter-crystalline weakness may result due to the liquid phase spreading over the crystal surfaces. The structure to be aimed at is one capable of deformation in any direction with the minimum of effort and the greatest possible freedom from the danger of cracking.

Most commercial alloys freeze over a temperature range and there is an increased tendency for shrinkage and gas porosity to occur, particularly where the freezing range is wide, unless solidification is directional toward the feeder head and, at the same time, rapid. Such alloys are also more susceptible to hot tearing.

Gas evolution during solidification is of two types: (a) Rejection of a gas (usually hydrogen) with a greater solubility in the liquid than in the solid metal, and (b) the formation of a compound gas, on solidification, by reaction between two constituents soluble in the molten metal. Whilst it is desirable to avoid gas pick-up during melting, it is usually necessary to adopt some method of removing dissolved gases.

Insoluble oxides, which might form films in the ingot, may be removed by agitating with a flux which dissolves, or at least wets, the oxide particles, and which has physical properties which allow it to separate readily from the melt.

Open slab casting is still extensively used in the non-ferrous metal industry, but the majority of ingots for working are cast in top-poured moulds. This method inevitably causes splashing and turbulence, and in order to avoid folds and to remelt splashes a high temperature and rapid pour are required. Air entrapment is also reduced by high pouring temperatures. On the other hand, to avoid shrinkage defects a slow pouring speed is required and a compromise of high temperature and low pouring speed is adopted. It is also common for a mould to be filled by a number of small streams of metal by the use of a tundish with a number of holes.

Copper, water-cooled, has now, to some extent, replaced cast iron for moulds for non-ferrous metals as it is not liable to develop crazing. Mould coatings are applied to reduce the initial chilling, to prevent adhesion and (sometimes) to provide a reducing atmosphere in the mould.

In cases where the metal has a great affinity for oxygen, some non-turbulent pouring method, such as the Durville process, has to be adopted.

The most important development of directional solidification is in the continuous and semi-continuous casting methods used to a limited extent for copper alloys and almost universally for aluminium alloys. The solidification is far from uni-directional and as a result high internal stresses may be generated, nevertheless these methods do approach more nearly to the ideal requirements than do any of the older methods of casting.

The macrostructure of ingots may have an important influence on subsequent working. The preferred orientation of columnar crystals may increase the difficulty of working metals with few planes of easy slip.

In alloys freezing over a temperature range, the structure tends to be finer and the columnar zone is largely or completely replaced by equi-axial grains. Small additions of certain elements may exercise a powerful grain refining action, but the mechanism has not yet been established.

THE PRODUCTION OF REFINED-COPPER SHAPES

By R. H. WADDINGTON.

THIS paper describes the procedure employed at a modern works for the production of standard and special shapes of electrolytic tough-pitch high-conductivity copper. Owing to difficulties inherent in reverberatory furnace melting of cathode copper, a changeover to electric-arc furnace melting was effected, resulting in a simplified process as no refining or slagging is necessary and optimum conditions can more easily be maintained. Melting is continuous in the 30-ton direct arc furnaces: two 256-lb. cathodes are charged per minute and molten metal is thereby displaced into 5-ton pour hearths, heated by L. F. induction. From the pour hearths, metal flows into single- or multi-lipped tilting ladles, and thence, either directly or through refractory funnels to the moulds.

By control of the electrolyte and the use of addition agents cathode copper is produced free from inclusions of electrolyte, slime, etc., which may act as gas-forming constituents in melting. In addition a controlled quantity of air is drawn through the furnace chamber to assist diffusion of gaseous products from the metal bath, and to impart to it an oxygen content of about 0.01%.

Pouring methods for both horizontally and vertically cast shapes are discussed and mention is made of the use of electrically-heated ladle lips where slow pouring is desirable. Copper moulds are used for all shapes, internal water cooling being applied for vertical casting. For tough-pitch copper, the mould dressing is a suspension of bone ash in water, whilst for phosphorus-deoxidised copper, the water is replaced by a heavy mineral oil. Automatic temperature control equipment is fitted to the pour hearth and mould temperatures are controlled by control of the cooling water temperature.

A comprehensive testing and inspection system enables the quality of the product to be checked against the conditions prevailing at the time of manufacture, so that modifications to the procedure may be made in line with the information obtained in this way.

MELTING AND CASTING ALUMINIUM BRONZE INGOTS FOR SUBSEQUENT WORKING

By A. J. MURPHY, M.Sc., F.I.M., AND G. T. CALLIS,
B.Sc.Tech., F.I.M.

THE main characteristics of the aluminium bronzes which are of interest in connection with the casting of ingots are their short freezing range, not exceeding 15° C., which accentuates the tendency for deep central piping, and the tenacious oxide film which forms on the surface of the molten metal and impedes the absorption of furnace gases and, when the metal is cast by non-turbulent methods, produces a very smooth skin on the ingot. The use of fluoride-containing fluxes is undesirable because, by removing the oxide film they allow gas to enter the melt and increase the risk of pin-holing.

The use of a non-turbulent pouring method, such as the Durville process, is essential in order to avoid

entrapment of oxide films in the ingot. The industrial application of the Durville process requires a robust machine to ensure steadiness of operation. Handling devices may be incorporated to release the machine for further pouring operations whilst the metal solidifies. Defects in Durville-cast ingots may arise from unsteady operation, insufficient skimming of the metal in the container, incorrect casting temperature, or excessive ratio of length to diameter, which should not exceed 8 : 1. Where ingots with a greater ratio are required vertical casting is used, with adverse effect on surface quality. Improvement may be achieved by special methods of casting which avoid excessive fall of the metal in pouring.

Accurate control of aluminium content is important in the range of composition used for hot forging and in this connection, hardness tests on the ingots provide a rapid check to supplement chemical analysis.

THE APPLICATION OF FLUX DEGASSING TO COMMERCIALLY CAST PHOSPHOR-BRONZE

By N. I. BOND-WILLIAMS, B.Sc., A.I.M.

FREQUENTLY the credit due to fundamental scientific work in the improvement of industrial processes is overlooked. In this paper the author shows that changes made in melting and casting bronzes in a British factory had their origin in published scientific work. The particular chill castings concerned were required to be suitable for cold working and had to fulfil certain technical, economic and commercial requirements.

In the original processes, melting was effected in a normal coke-fired crucible furnace, under charcoal. Phosphor-copper and then tin were added in the furnace and after skimming the metal was poured directly into cast iron moulds dressed with a heavy coat of seal oil and powdered French chalk or charcoal. As a result of a series of modifications made over a number of years a new procedure has been evolved. The furnaces have been rebuilt to reduce melting time and fuel consumption and to provide a strongly oxidising atmosphere. Before charging, a slag is prepared from sand, copper mill scale and borax. Tin is added while the crucible is still in the furnace, but the phosphor copper is not added until the crucible has been withdrawn and the slag skimmed off. Pouring is effected by the use of a tilting cradle and a tundish with a graphite-bush outlet. The moulds, which have a heavier base to encourage solidification from the bottom, are dressed with a volatile oil without solid addition.

As a result of the modified procedure, the castings are more consistent in size and shape, the tendency for a deep central shrinkage cavity is reduced, feeding is facilitated and the physical and chemical properties are improved, largely as a result of the reduction in unsoundness, which obstructs the absorption of the tin-rich constituent into solid solution by heat treatment. Tin sweat, which necessitated machining of the ingot surfaces, is no longer encountered. The tin-rich phase does not occur in such large areas, and by reason of the reduced lateral shrinkage, it is not exuded between the cored crystals into the space between the surface of the casting and the mould. Internal gas pressure is reduced by the de-gassing process and this also reduces the tendency for exudation. Internal soundness is also improved.

Over a period of ten years a considerable improvement in technical properties, costs and commercial advantages has been achieved as a result of the application of principles established by scientific research.

THE MELTING AND CASTING OF BRASS

By M. COOK, D.Sc., Ph.D., F.I.M., AND N. F. FLETCHER,
A.I.M.

WITH few exceptions brass alloys are easier to produce in the form of chill-cast shapes than any other major group of copper-base alloys, provided advantage is taken of the available knowledge and experience. In many specifications the permissible limits of impurities are higher than fabrication requirements can tolerate; on the other hand certain impurities may be beneficial as regards fabrication. It will be seen, therefore that within the specification limits, the composition can be all-important in determining the suitability for a particular fabricating process.

Since the alloys are not, in general, sensitive to gas pick up, because of the high vapour pressure of zinc, nor do they suffer much more than loss of zinc by oxidation, the choice of melting unit is determined largely by economics, type and quantity of production, availability of fuels and the like. For large outputs of a particular composition the low-frequency induction furnace is largely favoured. For smaller quantities of a variety of compositions, the various types of crucible furnaces and electric arc furnaces of the rocking type find application. With good melting practice, the risk of contamination from furnace gases and crucibles is negligible. Stirring rods should be coated with a refractory wash or sleeve to prevent iron pick-up. In this connection the self-stirring action of the metal in the L.F. induction furnace is of advantage, as it is also in ensuring efficient mixing of added alloying elements. Due to its high vapour pressure, at elevated temperatures, in both the liquid and solid state there is a steady loss of zinc unless prevented by a charcoal covering. In melting alloys containing such readily oxidisable elements as silicon and aluminium, oxidation should be kept at a minimum since oxides of these elements, if they enter the mould are liable to cause defects in the casting.

Cast iron moulds are still extensively used in the wrought brass industry, but their marked surface cracking tendency, which gives rise to blowing defects in the surface (as a result of imperfect drying of mould dressings) poor ingot surfaces and, in the extreme, cracked ingots, has resulted in increasing use of copper-faced water-cooled moulds. Except in the case of special pouring and mould arrangements designed to promote tranquil conditions, when non-flaming dressings are preferred, flaming dressings are used because of the action of the reducing atmosphere, which they produce, in protecting the metal stream from oxidation.

Vertical moulds are now generally used in brass casting, the metal being poured into a runner box or tundish. In the casting of brasses containing aluminium, non-turbulent methods such as the Durville process are used, although, with suitable precautions direct pouring via a tundish can be used with a consequent reduction in grain size.

Segregation, although not a major problem in brass alloys, does occur with certain compositions, with resultant difficulties in fabrication, and consequently pouring conditions have to be carefully controlled to minimise the effect.

THE MELTING AND CASTING OF NICKEL SILVER AT THE WORKS OF MESSRS. HENRY WIGGIN & CO., LTD.

By E. J. BRADBURY, M.Eng., A.M.I.Mech.E., A.I.M., and P. G. TURNER, Assoc.Met.

BEFORE the war, the melting plant consisted of 30 coke-fired crucible furnaces in six shops of five furnaces each, but in 1940 a battery of 12 lift-out-type oil-fired furnaces was installed in a new melting shop. Subsequently one of the five-furnace shops was converted to two coke-fired and two oil-fired furnaces, and produced as much metal as a shop with five coke-fired furnaces. Silicon-carbide has replaced graphite for crucibles as its lower thermal conductivity helps to keep the metal hot and reduces the temperature difference between the first and last ingots poured. More heats per crucible are obtained than with graphite crucibles and the life has been further extended by using them for high-grade high-nickel alloys when new, and subsequently for low-grade low-nickel alloys.

Split cast iron moulds were used initially for all types of ingots, but experiments with water cooled moulds led to their use for all strip ingots. A mixture of rape oil and dust charcoal is used for dressing the moulds.

Nickel shot, copper, zinc and scrap are charged and the red hot crucible and any excess scrap is charged as space becomes available on melting. The decision as to when the metal is ready to pour is left to the judgment of the caster. *Borocalcite* is thrown in as a flux on charging. The change to this material from borax/boracic acid and mixtures has reduced the flux-line attack on the crucible. In spite of the large amount of zinc in these alloys, deoxidation has been found to be advantageous. One ounce manganese and $\frac{1}{2}$ oz. phosphor-copper (10%P) are used per 50 lb. of metal. The phosphor-copper addition was developed as a result of difficulties that were experienced with production of metal for spinning and pressing, in that the metal tended to crack during annealing.

The metal is poured into the vertical moulds via a tundish. When the ingot is ready for feeding, the tundish is removed and the ingot is fed through the charcoal dust covering which is then added. Wirebar ingots are fed through a firebrick nozzle. Extrusion-billet ingots are poured and fed through a large $5\frac{1}{2}$ in. dia. riser made of red moulding sand.

The oil-fired furnaces produce metal for a cost which is less than that for the coke-fired furnaces by one-tenth of a penny. Production rate is, however, higher and this was extremely useful during the war. From a production aspect, the most outstanding factor has been the introduction of the water-cooled mould. Not only was production increased thereby, but the working conditions were greatly improved as a result of the decreased shop temperature.

Joint Discussion

The successful conduct of the discussion on the papers prepared for the Symposium owed much to the efforts of PROFESSOR O'NEILL in his role of Rapporteur. The papers were not presented by the authors, but in his introductory remarks PROFESSOR O'NEILL referred to some of the salient points of the various papers and indicated aspects on which discussion would be of benefit. As a link between the morning and afternoon sessions PROFESSOR O'NEILL opened the latter with a resume of the morning discussion and finally

rounded off the meeting with a brief summing-up. In the notes which follow we have endeavoured to digest the main points arising in the discussion.

Although PROFESSOR O'NEILL's suggestion that the thermodynamic aspects might be considered was ignored, a number of speakers were prepared to discuss other theoretical aspects of the subject. DR. N. P. ALLEN drew attention to the behaviour of metals towards gases in relation to their position in the electrochemical series. On passing from the noble metals to the base metals the origin of gaseous trouble changed from oxygen to hydrogen as the stability of the oxide increased, with an intermediate region containing copper, iron, nickel, etc., in which the oxide was not very stable and where trouble arose from a reaction between the oxide and some non-metallic impurity such as hydrogen, carbon or sulphur. He felt it was necessary to study the chemistry of molten metal in order to determine the factors which had to be controlled. Only by the complete understanding of all the factors involved could such a plant as that described by Mr. Waddington have been put into operation.

On the physical aspects of gas evolution, MR. E. A. G. LIDDIARD pointed out another virtue of tranquil pouring, namely the preventing or delaying of the initiation of bubbles of dissolved gas, which, MR. A. G. RAMSEY emphasised, must be hydrogen as no other gases dissolved in molten metals to any appreciable extent. On the other hand MR. LIDDIARD quoted a foundry where molten aluminium was always allowed to stand after chlorine degassing as otherwise gas unsoundness was experienced.

MR. W. F. BRAZENER referred to the work of Dr. Allen on the effect of dissolved hydrogen in copper and pointed out that in his paper on the flux degassing of phosphor bronze, Mr. Bond-Williams demonstrated the practical application of Dr. Allen's results by using an increase in oxygen content for degassing. MR. R. W. RUDDLE queried a statement in the first paper to the effect that the degassing of aluminium alloys was more readily effected with chlorine than nitrogen. In his view they were about equally effective for most alloys, but for magnesium containing alloys, nitrogen was definitely superior because of the action of chlorine in forming liquid magnesium chloride instead of gaseous aluminium chloride. DR. G. BLANC spoke of experiences in France with gas unsoundness in copper alloys. They preferred the use of an oxidising atmosphere to nitrogen scavenging, although he quoted improved results with a high-lead tin-bronze using nitrogen. DR. BLANC thought the mechanism might be an oxidising one resulting from the contact of the greater part of the metal with the oxidising atmosphere as a result of the energetic bubbling of the nitrogen.

MR. F. C. EVANS and DR. RAMSEY raised the question of the healing of gas holes by pressure welding during hot working, the former suggesting that if the mechanism were more clearly understood, it might be possible to make flat-topped ingots of high shrinkage alloys by control of the gas content. DR. RAMSEY suggested that success with tough pitch copper where the gas was steam, which could not diffuse away, was due to the rolling pressure reversing the reaction with the production of cuprous oxide and hydrogen, which could diffuse away.

Most of the references to melting equipment concerned electrical methods—high and low frequency induction and arc furnaces—although MR. G. SKRIPT put forward the claims of the rotary furnace as well. Even the charge of high costs which is often levelled against electric melting

was refuted by MR. D. F. CAMPBELL, at least for a new plant where such auxiliaries as chimneys would be needed for fuel-fired furnaces. Several speakers were surprised that crucible melting was employed for nickel silver, DR. O. C. H. MESSNER stating that in Switzerland they had made extensive trials on the high and low frequency methods and had decided in favour of the latter, which lined up with British practice as described by MR. E. A. BOLTON. DR. MESSNER said they had used linings developed for melting zinc alloys by this method with a substantial increase in life. MR. CAMPBELL and MR. P. F. HANCOCK pointed out the advantage of electric melting in avoiding gas pick-up and MR. HANCOCK referred to the curious fact that the most widespread adoption of electric melting had been for brass where gas pick-up was of no moment. A further advantage of induction melting, particularly at high frequency, was the efficient mixing action caused by the movement of the metal, which MR. EVANS had found to be extremely valuable in melting aluminium bronze. The low frequency induction process was not suitable for small batches of a variety of materials and in such cases the rocking arc furnace came into its own.

MR. BOLTON asked whether the authors practice was to charge the zinc with the other metals in melting nickel silver, as seemed to be indicated, because such a procedure would seem likely to result in increased zinc loss. In referring to the same paper, MR. EVANS was rather surprised at the use of silicon carbide crucibles as his experience suggested that carbon was more readily picked up from them than from graphite crucibles. On the question of crucible life, MR. A. L. MOLINEUX stated that increased life could be obtained by using them for phosphor bronze for the first twenty-five heats and then using them for brass.

Tundish pouring seemed to have many advocates, although MR. C. SMITH considered that dirt in the casting was an inevitable result. MR. CAMPBELL referred to the use of a tundish in a controlled atmosphere, to protect the metal stream, which he had seen in America.

Although such materials as tough pitch copper and 60/40 brass were suitable for open flat-type mould casting, MR. W. F. BRAZENER pointed out that the vertical mould was being increasingly used in the non-ferrous industry. The greatest step forward was the introduction of water-cooled moulds which, despite their higher cost, were cheaper per lb. of metal cast, than cast iron moulds, apart from the question of scrap due to bad surfaces. MR. H. DAVIES drew attention to the effect of water-cooled moulds in preventing the occurrence of blistering in copper sheet, possibly as a result of the rapid cooling preventing coalescence of gas into large bubbles and MR. EVANS described cylindrical cast iron cannon type moulds to which he fitted a block copper base which helped solidification from the bottom.

The vexed question of mould dressings came in for a good deal of attention. MR. DAVIES criticised the criteria postulated by Mr. Waddington for a satisfactory mould dressing. Judged by those standards, lime and certain clays would be ideal dressings, whereas in actual fact there did not seem to be any substitute for bone-ash. MR. L. NORTHCOTT was surprised that flaming dressings were still in use in this year of grace 1949 in view of the findings of Mr. Bailey, a quarter of a century ago, that flaming dressings gave rise to sub-surface cavities leading to spills. A soot dressing with externally applied reducing atmosphere obviated the defects. As flaming dressings had been quoted as refining the grain of the ingot, MR. BRAZENER wondered

whether the effect was due to the fact that with a non-flaming dressing, a deposit was left on the mould surface, thus hindering cooling or rather to particles of combustion products becoming trapped in the metal and acting as crystallisation nuclei. DR. MESSNER said they used flaming dressings exclusively for water-cooled moulds. He also mentioned that in America soda ash was added to mould dressings and in a Continental works resin was used. Insufficient attention was paid to the oily-vehicle forming part of the make-up of mould dressings in MR. W. R. BALLARD's view. As far as creating a reducing atmosphere was concerned there was probably little to choose between various oily substances, but their physical properties insofar as they affected the mechanical functions of the vehicle were of primary importance. After trying aluminium paint, without too much success, MR. MOLINEUX said they had reverted to French chalk and tallow.

Several speakers referred to the usefulness of the Durville process in securing tranquil pouring. The process was not without its difficulties; as MR. CALLIS pointed out, it was very difficult to get a Durville ingot free from axial unsoundness due to the tendency for bridging to occur. MR. EVANS and MR. SKRIFT emphasised the importance of careful tilting of the machine through the horizontal when the centre of gravity changed from one side to the other. M. LOISEL said they were using the Durville process for sand castings, taking precautions with cores to ensure that the pressure of the metal did not displace them during tilting.

Directional solidification was discussed by MR. RUDDLE who showed that rapid pouring would tend to axial unsoundness and that for the best results the mould should be designed for the maximum removal of heat from the bottom and the slowest practicable rate of pouring should be adopted—both conditions employed in the direct-cooled continuous casting process for aluminium. MR. F. HUDSON described a casting method patented by Willcox in America, in 1933, in which the metal was melted in a high frequency furnace and then poured from it to the mould in a manner similar to the Durville process, the mould also being heated by high frequency induction. In this way equi-axed crystals could be produced, and by allowing the mould to fall slowly through the coil, solidification from the bottom could be encouraged.

Both in the papers and the discussion references were made to continuous casting, but MR. H. W. L. PHILLIPS issued a warning against light-hearted adoption of the method. The chief drawback was the development of high values of internal stress which might result in hair cracks, with trouble on subsequent fabrication due to their opening up, or in extreme cases, splitting of the block from end to end. Even if cracking did not occur during cooling there was no certainty that it would not on scalping or sawing to length. It would be seen, therefore, that considerable development was necessary to ensure success. Again the effect of casting conditions on the structure as it affected subsequent working and heat treatment should be considered. DR. J. W. CUTHBERTSON produced samples of bronze bar which had been made by a semi-continuous casting process which was capable of producing cored or solid bars.

DR. GENDERS referred to the casting of small slugs for direct working and to the Soro process in which a ring was centrifugally cast with a feeder head which was subsequently turned off. After cutting, the ring was straightened and either worked as one piece or cut up for forming.

Final Session

THE RECOVERY AND RECRYSTALLISATION OF ROLLED ALUMINIUM OF COMMERCIAL PURITY

By P. C. VARLEY, M.B.E., T.D., M.A.

IN the experiments described in the paper, an attempt is made, using polycrystalline aluminium of normal commercial purity, to bridge the gap between recovery and recrystallisation and to determine whether they are in fact two distinct processes, each with its own activation energy, or whether they are merely different stages of a single process, with but a single activation energy.

The progress of recovery and recrystallisation was followed by tensile tests after varying periods of heating to various temperatures in the range 200°–325° C. Preliminary experiments showed that small variations in silicon and iron content were without influence on the rate of softening. Chill-cast and continuously-cast material likewise behaved similarly. Plotting U.T.S. against the logarithm of time curves, are obtained for various temperatures, which are superposable by displacement along the time axis. One set of samples had received an intermediate anneal during rolling and the curves for these samples were superposable on the curves for material without process anneal by displacement along the U.T.S. axis.

In order to determine the point on the softening curves at which the first signs of recrystallisation could be detected, X-ray diffraction patterns were prepared by the transmission method. The degree of recrystallisation was assessed on an arbitrary scale and plotted against tensile strength. From the curve it appears that recrystallisation, as distinct from recovery, sets in at 7½ tons/sq. in. No evidence was obtained of any changes in structure during the purely recovery part of the process.

The form of the isothermal annealing curves show no sign of the changeover from recovery to recrystallisation. Furthermore the author has determined the activation energy of the process and found it to be the same for both recovery and recrystallisation, and it is suggested that the two processes are similar in their nature. Both probably involve the diffusion of dislocations through the lattice and the annihilation of those of opposite sign, the additional activation energy involved in the formation of new crystals being too small to be detected by the methods adopted.

Discussion

DR. T. LI. RICHARDS in opening the discussion said that although there were many interesting points raised in the paper, he would confine his remarks to the main objective of the investigation which was to determine whether recovery and recrystallisation were one and the same process. He thought the evidence was in favour of the view that the processes were distinct and separate. DR. RICHARDS considered that the isothermal curves showed evidence of two distinct processes by a change in slope, but he pointed out that the author would have obtained two structural changes had he used the X-ray back reflection method rather than the transmission technique. In DR. RICHARD's view, recovery, the development of mosaics and polygonisation should all be regarded as the same thing.

MR. R. J. L. EBORALL differed from DR. Richards in that he thought the recovery processes might well prove

the key to the difficult feature of the delay in the formation of the nuclei on which recrystallisation depends. In some X-ray work on recovery they had found the first stage to be removal of the line broadening, followed by fragmentation so that maxima developed in the reflections, eventually becoming more or less distinct spots. It seemed that different fragments became perfect at different rates and it was possible that those which became perfect first acted as recrystallisation nuclei.

In his reply MAJOR VARLEY showed some slides of curves obtained on an aluminium alloy containing 2% magnesium, in which the activation energies for recovery and recrystallisation were not the same. It was evident that the question would only be settled as a result of many people tackling the problem along different lines.

HIGH-TEMPERATURE TENSILE PROPERTIES OF CAST ALUMINIUM-SILICON ALLOYS AND THEIR CONSTITUTIONAL SIGNIFICANCE

By W. I. PUMPHREY, M.Sc., and P. H. JENNINGS, Ph.D.

THE tensile properties at high temperatures of some aluminium-silicon alloys were determined (a) during cooling from the liquid state, and (b) after rapid heating of chill-cast alloys. Strength-temperature curves relating to both conditions were obtained for ten alloys containing from 0-12% silicon.

The curves consist of a relatively low-temperature portion in which the alloys are ductile and in which the strength falls gradually as the temperature is increased, and a steeper high-temperature portion in which the alloys fracture in a brittle inter-crystalline manner until a temperature is reached when so much liquid is present in the mass that coherence is lost.

The point of discontinuity in the curve was found to be associated with the effective solidus temperature, and the position of the point of non-coherence to be a function of the proportion of liquid in the alloy. The interval between these temperatures—i.e., the brittle range was correlated with the tendency to cracking during casting and welding, the greatest susceptibility to cracking being found in those alloys in which the temperature range in which brittle fractures occur is most extended. With any alloy, the brittle range is less, as determined by tensile testing after rapid heating from the solid state, than by testing during cooling from the liquid state. The difference is ascribed to differences in crystal size rather than to the different thermal conditions obtaining in the two tests.

A CONSIDERATION OF THE NATURE OF BRITTLENESS AT TEMPERATURES ABOVE THE SOLIDUS IN CASTINGS AND WELDS IN ALUMINIUM ALLOYS

By W. I. PUMPHREY, M.Sc. and P. H. JENNINGS, Ph.D.

PREVIOUS papers have described the tendency of many aluminium alloys to crack at temperatures above the solidus during casting and welding. In the present paper, a critical examination is made of the cause of such cracking.

Cracking at temperatures above the solidus occurs in the brittle temperature range between the effective solidus temperature and the temperature at which a coherent dendritic structure is first formed in the alloy during cooling from the liquid state, and is a result of contraction stresses. The inherent tendency of an alloy to such cracking (shrinkage brittleness), is directly

proportional to the extent of the brittle range and to the contraction that occurs during cooling through this range, and is independent of all external factors, except those which alter the extent of the brittle range—i.e., those which modify the course of solidification. The extent of the brittle range is primarily determined by the constitution of the alloy in the relevant conditions, but it is also dependent on the amount and mode of distribution of the eutectic liquid within the mass. A minimum shrinkage-brittleness in any particular alloy is associated with conditions in which there is no tendency for entrapment of eutectic liquid between the growing dendrite arms during solidification, and when, in consequence, all the eutectic in the finally solidified alloy is situated at the crystal boundaries. The degree to which liquid is entrapped between the dendrite arms during their growth is a function of the linear rate of crystal growth, which in turn is dependent on the rate of cooling and the final crystal size and on such secondary factors as gas content and the effect of alloying additions on the dendrite configuration.

A characteristic curve relating shrinkage-brittleness with rimosity (actual severity of cracking in particular circumstances) can be derived but the precise form is governed by the sensitivity of the test. The relationship may be modified in some measure if the alloy is capable of accommodating the contractual stresses in the brittle range by a process involving minor adjustments in the relative positions of the crystals. Rimosity is also dependent on the size and shape of the cracks which occur and on the external stresses to which the solidifying alloy is subjected.

A mathematical relationship has been formulated which enables an estimate to be made of the manner in which weld cracking, in a butt weld between two restrained sheets of aluminium alloy, is likely to be affected by alteration in the physical properties of the parent metal or by modification of the welding technique. The relationship indicates that the likelihood of cracking is increased by increase in the extent of the brittle temperature range, speed of welding, rate of cooling of the weld, the degree of restraint, the thermal conductivity, or the linear coefficient of thermal expansion of the parent metal.

A CONSIDERATION OF THE NATURE OF BRITTLENESS AT TEMPERATURES BELOW THE SOLIDUS IN CASTINGS AND WELDS IN ALUMINIUM ALLOYS

By W. I. PUMPHREY, M.Sc., and D. C. MOORE, B.Met., M.Sc.

CRACKING at temperatures below the solidus has been observed in a number of aluminium alloy systems. The occurrence of such cracking depends on the ductility of the metal, on the conditions of external and internal stress to which a cooling metal is subjected at sub-solidus temperatures, and on the amount of cracking which occurs at temperatures above the solidus during the cooling of the metal from the liquid state.

Alloys which crack at temperatures below the solidus in the restrained-weld test do not invariably so behave in the ring-casting test. The difference has been ascribed to the pronounced stress-raising effect of the notch at either end of the weld bend in specimens tested in the restrained-weld test.

It is not considered that sub-solidus cracking will normally be encountered in industrial conditions of

casting and welding, except where alloys of low ductility are exposed to severe contractual restraint. Sub-solidus cracking may, however, be encountered during the cooling of castings which are large in size or complex in form if the cast metal is of low ductility. In such instances, the degree of sub-solidus cracking which occurs may be regulated by control of the factors mentioned in the first paragraph.

Joint Discussion

In opening the discussion, DR. E. G. WEST said that the work was approaching the conclusion they had hoped for at the outset, and the results now required to be applied by the industrial users of materials. In a written contribution presented by MR. J. C. BAILEY, MR. D. C. G. LEES said he was pleased to note the wide measure of agreement with his own work on hot tearing. They both agreed that the danger of tearing only arose when a coherent network had been formed by the growth of the interlocking arms of the dendrites. Whilst the constitutional factors were no doubt the most important in determining the incidence of hot tearing, two minor ones, grain size and gas content should be considered and further study of these factors would be desirable.

MR. W. A. BAKER, in a written contribution presented by MR. R. W. RUDDLE, commented on the healing of hot tears. Whilst with the large hot tears considered by the authors healing might be rare, such healing did occur in micro-tears during the solidification of castings. Speaking generally, the tendency of an alloy to hot tear was a good measure of its susceptibility to mechanical weakness if its freezing shrinkage were imperfectly fed. The authors' views on the occurrence of the healing effect would preclude that useful generalisation.

In replying to the discussion MR. W. I. PUMPHREY said that whilst they did not consider that healing of small tears did not occur, they felt that it occurred less frequently in small castings in which there was no reservoir of healing liquid. The questions of grain size and gas content, raised by MR. LEES, would, he hoped form the subject of future work.

THE MEASUREMENT OF GRAIN-SIZE OF TUNGSTEN AND TUNGSTEN CARBIDE POWDERS USED FOR THE MANUFACTURE OF HARD-METAL

By H. BURDEN, B.Sc., and A. BARKER, B.Sc., A.R.C.S., A.Inst.P., L.I.M.

THE hardness of tungsten carbide-cobalt hard metals is, in the main, governed by the amount of cobalt present and the grain-size of the tungsten carbide phase in the finished metal. The latter is in turn influenced by the particle size of the original tungsten powder and grain growth during processing. The importance of accurate measurement of the grain-size of the powder will be appreciated and in this paper the authors discuss various methods of measurement.

The sedimentation balance was abandoned at an early stage in the work due to the time required to carry out a test and to the difficulty in obtaining reproducible results. The turbidimeter, in which the amount of light passing through a water suspension of the powder is measured after various times of settling, gives the size distribution of the particles. Whilst the test time is reduced compared with the sedimentation balance it is still rather long for a works test. A method using the Spekker absorptiometer has been developed which, whilst measuring the specific surface, from which

the surface mean-diameter can be deduced, rather than the size distribution, has given results which show good correlation with values determined by microscopic methods. As it is a rapid method (10 minutes for a complete determination) it suffers less from errors due to aggregation than do the previous methods. For particle shape studies microscopic methods are most suitable.

It is concluded that of the methods discussed none is suitable for accurate size-distribution measurements on the types of powder tested. For a rapid method, the most promising direction for further work seems to be the development of an improved turbidimeter, coupled with a greater knowledge of aggregation sedimentation phenomena, and light absorption by particles.

For the time being, the Spekker method is used as the basic means for measuring particle size, supplemented where necessary by microscopic examination for shape and to indicate size range.

Discussion

The discussion was opened by MR. E. A. OLIVER, who emphasized the difficulties of grain-size measurements. It did not appear that the major problem of aggregation had been solved and until a solution was found, measurements could never be more than a rough guide. Particles below 0.2 micron were of the utmost importance in grain growth on sintering but many of the usual methods ignored them or gave inaccurate figures.

MR. OLIVER did not consider the sensitivity of the method had been seriously tested by using four widely varying samples. The most promising technique was electron-microscopy, but there did not seem much likelihood of its development as a routine control.

MR. E. VAN SOMEREN suggested that ethylene glycol should be used as a suspension medium in the hope that aggregation could be reduced. Another possibility was to heat a small quantity of powder in oxygen when the gain in weight should be a characteristic of the specific surface.

In reply MR. A. BARKER said the samples were chosen with widely varying sizes because they wished to get a definite correlation between particle size and hardness of the sintered material. In point of fact, they had been able to measure quite small differences by the Spekker method. They had used ethylene glycol but there was difficulty in getting rid of air bubbles. On the whole water was much more satisfactory. The gain in weight on oxidation method might be of value for tungsten.

Staff Changes and Appointments

MR. N. G. LANCASTER, M.B.E., A.C.A., has been elected a Director of Tube Investments, Ltd. MR. ROBERT A. STAUFFER, has been elected Vice-President and Director of Research of National Research Corporation, Cambridge, Massachusetts. Mr. Stauffer, a graduate of Harvard University, has been associated with the company since 1942.

New Fellows of the Royal Society

Among recent elections to Fellowships of the Royal Society were those of:—
Dr. R. W. BAILEY, Consulting Engineer, Research Dept., Metropolitan-Vickers Electrical Co., Ltd.
Dr. U. R. EVANS, Reader in Metallic Corrosion, Cambridge University,
Sir BEN LOCKSPEISER, Chief Scientist, Ministry of Supply.

MICROANALYSIS

CHEMICAL AND PHYSICAL METHODS

APPARATUS

METALLURGICAL APPLICATIONS

TECHNIQUE

PUBLICATION of the First Annual Review of Analytical Chemistry prompted us to give some thought to the changing face of this science. Analytical Chemistry may be said to be a little more than a century old. But on scanning the index in the Fundamental Analysis section of the review, it is noteworthy how many of the topics would, little more than ten years ago, have been missing from the record, or only just have been considered worth including. Light and ultra-violet absorption spectrophotometry had been long established, though the methods have changed radically. Emission spectroscopy and light microscopy also date from the classical period. General polarography and chromatographic analysis had just received a considerable amount of publicity, but would have been relative newcomers. Electroanalysis, distillation, extraction, indicators, inorganic and organic microchemistry, gravimetric and volumetric analysis could all, presumably, expect to be included. This gives a total of sixteen of the present sections, or a little over half. On the border-line between inclusion and non-inclusion we have infra-red and Raman spectroscopy, X-ray diffraction analysis, organic polarography, amperometric titrations and ion exchange. Such procedures as X-ray adsorption, mass spectrometry, electron microscopy, nucleonics and fluorimetric analysis have had their main development within the past ten years. It is doubtful if instrumentation would have been accorded separate consideration, and almost certainly statistical methods in analytical chemistry would have been passed over. To this simple survey we must add the fact that the one hundred and seventy-odd pages of the review—we are still considering only the fundamental section—are primarily concerned with developments during the past five years only. We are thus presented with a formidable and rapid development of which we have all been, in a sense, aware, but which can only be fully brought home by a survey such as this.

Miscellaneous Microchemical Devices—XVIII

Two Simple Microburettes

By J. T. Stock and M. A. Fill

(L.C.C. Norwood Technical College, London, S.E. 27).

DURING the course of our experiments, a variety of simple microburettes have been developed; two of these are described below.

The microburette shown in Fig. 1 incorporates several of the features of the one previously described.¹ It is filled by opening the control valve at the head of the burette, immersing the tip in the requisite solution, and sucking at the side tube. Flow is stopped by turning the knob of the control valve through 180° in either direction.

Construction of the parts of the control mechanism is shown in Fig. 2. Two pieces of thin-walled brass tubing of diameters to telescope into one another are needed (old cork borers are excellent). The tubes are cut squarely, the narrower (a), to a length of about 5 cm. and the wider (b), to about 3 cm. long. Using a hacksaw with a fine blade, the wider tubing is cut obliquely as shown at (b). About 10°–15° from the transverse section is the correct angle. The oblique portions are then slipped on to the narrower tube, and one portion is

secured by soldering as at (c). The knob attached to the other end of (a) is a bakelite cap from an ink-bottle which has been filled with sealing wax. While the latter is still molten, the warmed end of the tubing is thrust in and held squarely until setting has occurred. A small glass or metal plunger (d), of diameter to slide easily but without undue shake in the body of the microburette is also constructed. At its lower end, the plunger carries a cylindrical plug of soft rubber which has been cut with a sharp cork borer. The outer face of the plug should be truly at right angles to the axis of the plunger.

By means of sealing wax, the portion of the obliquely-cut tube nearest the knob is secured in one end of the tee-piece forming the body of the microburette, care being taken that the mounting is truly axial. The plunger is then dropped into place and the rubber sleeve on the burette stem is wetted and forced into the body. Before mounting the burette stem, the end should be ground flat and at right angles to the axis of the stem. The position of the upper end of the burette stem should

¹ Stock, J. T. and Fill, M. A., *Metallurgy*, 1944, 31, 103.

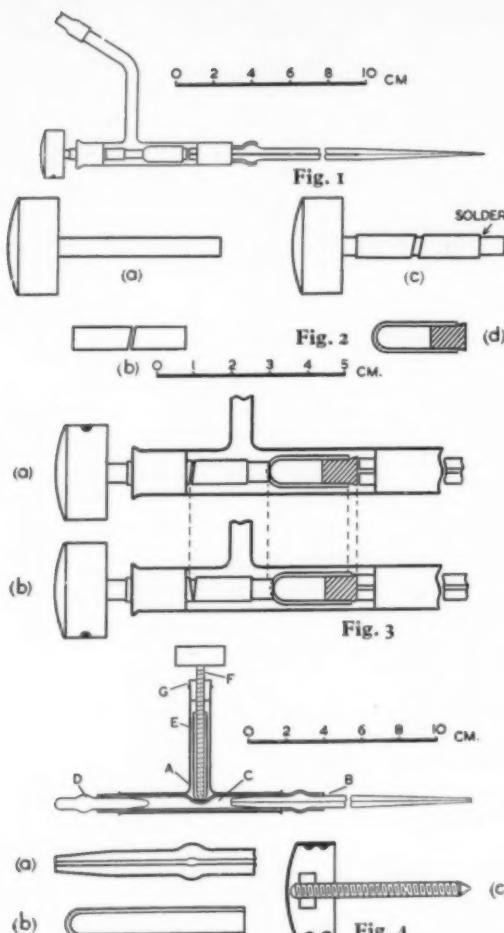


Fig. 1.—Cam-action microburette. Fig. 2.—Construction of parts of control mechanism. Fig. 3.—Principle of control mechanism. Fig. 4.—Compression-type microburette.

be adjusted so that, with the oblique portions of the brass tubing mated as shown at (a) in Fig. 3, the face of the rubber plug is just clear of the end of the burette stem. Rotation of the knob through 180° then causes the plug to be forced by cam action on to the end of the burette stem, as shown at (b), thus arresting the flow of liquid from the burette.

Liquid is expelled from the microburette shown in Fig. 4 by compressing the air within a sac formed from a length of rubber tubing.² The body A consists of a tee-piece with arms of equal bore. Attachment of the interchangeable stem B is effected by a sleeve of rubber tubing which also forms sac C. In order that the rubber tubing may easily enter the body and yet form a tightly-wedged joint between body and stem, the latter is thickened up near the end and then tapered, as shown at (a). The sac, which should be cut to project about 1 cm. from the other end of the body, is closed by a tapered plug D of glass rod. This is wetted and forced in so that the sac is neither stretched nor compressed. Plunger E, shown enlarged at (b), is of glass tubing, or, much better, of brass. It should slide easily, but without undue shake, in the side arm of the body. Operating screw F, which is of 4 BA brass screw rod, has a rounded point and should slide freely within the plunger. The screw passes through a cylindrical nut G (e.g., a terminal head) cemented in the side arm. To key the screw to the sealing wax with which the knob is filled, an ordinary brass hexagon nut is screwed on for a few turns before forcing the screw into the molten wax, as shown in section at (c).

On rotating the knob in a clockwise direction the sac is compressed and air is expelled. By dipping the tip of the burette stem into the requisite solution and rotating the knob in the opposite direction, liquid is drawn in, and may be expelled in very small portions as required.

Either type of burette may be affixed to a board carrying a millimetre scale, or, preferably, the rapidly-detachable scale unit previously described may be used.¹ In the latter case, the burette assembly may be held in an ordinary retort clamp.

² Holt, P. F. and Callow, H. J., *Jour. Soc. Chem. Ind.*, 1942, 61, 99.

Group Separations in Inorganic Qualitative Analysis

Part II—Sulphide and Potential Sulphide Ion Reagents*

By Francis R. M. McDonnell and Cecil L. Wilson

Microchemistry Laboratory, Chemistry Department, The Queen's University of Belfast.

Vortmann¹⁶ has devised a different method of forming the sulphides, by fusion of the material to be analysed with sodium carbonate and sulphur. Separations are achieved by extraction with water, and further fusion of the reprecipitated sulphides with potassium pyrosulphate. The scheme, shown in Table VII, includes several of the rarer elements, thallium, uranium, beryllium, molybdenum, vanadium, selenium, tellurium and gold being placed in it.

* Concluded from March issue, page 280.

Miscellaneous Sulphur Compounds

Several authors, ^{17, 18, 19, 20, 21}, have put forward schemes based on the use of thioacetic acid or its ammonium salt as a substitute for hydrogen sulphide. By heating thioacetic acid to 90° C. with mineral acids, when it is decomposed to hydrogen sulphide and acetic acid, precipitation of the cations of the normal sulphide group can be affected, even in the presence of a large excess of mineral acid, and particularly under pressure.²¹ Zinc is completely precipitated at 90° C. in a sealed tube

if the free acid concentration does not exceed 3.5%. Cadmium, nickel, cobalt and iron are also precipitated, while manganese remains in solution.

Again, the sulphides of arsenic, antimony and bismuth are soluble in excess of the reagent, with the formation of addition compounds, such as arsenious thioacetate, and the liberation of hydrogen sulphide.

Since the only decomposition products are acetic acid and hydrogen sulphide,¹⁸ the analysis of later groups is not interfered with, and the time for precipitation is very considerably lessened.

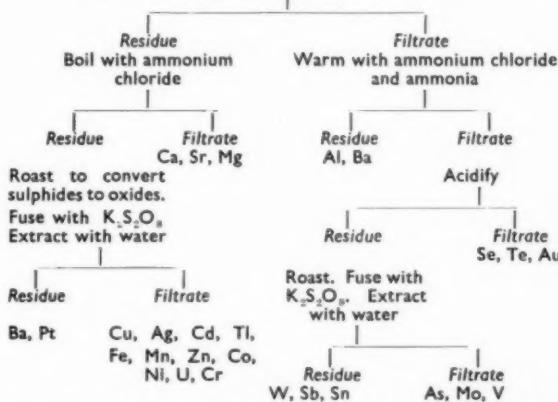
Among other reagents which have been proposed, ammonium dithiocarbonate has been advocated by Vogel²² but it cannot be used as a universal substitute for hydrogen sulphide or ammonium sulphide. Geddis² has tried a number of reagents containing a potential sulphide ion, including thioacetic acid, thiourea, and sodium diethyldithiocarbamate, all of which proved unsuitable for unspecified reasons.

Thiosulphates

Probably the first alternative to sulphuretted hydrogen to be proposed, from the historical point of view, was sodium thiosulphate, which was advocated by Hinley²³ in 1842, and this reagent has been used in several schemes proposed at intervals from that time to the

TABLE VII

The material for analysis is fused with sodium carbonate and sulphur, and the fusion product is extracted with water



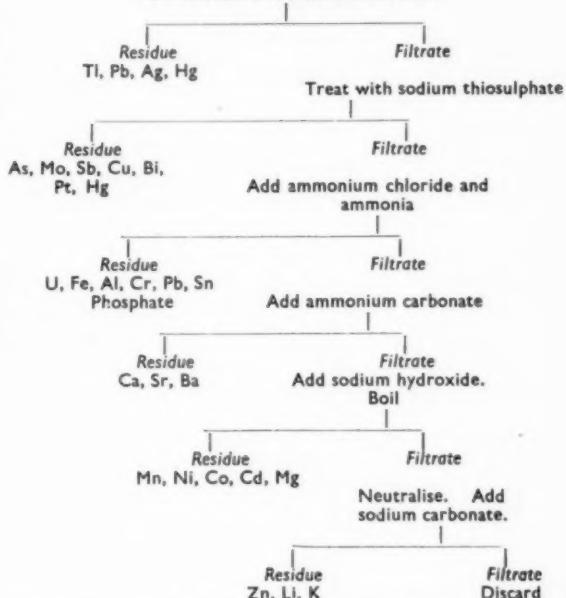
present. Sodium thiosulphate will precipitate the sulphides of copper, mercuric mercury, bismuth and arsenic from hot, slightly acid solution. According to Mee²⁴ the reactions occurring appear to be as follows, taking copper chloride as the specimen substances:



Vortmann²⁵ states that for maximum precipitation the solution must not contain too much free acid, and no nitric acid, although no definite ideal value for the acid concentration is given. In addition, he recommends that the reagent be added in small quantities at a time, and not in excess. In his scheme, Vortmann precipitates Group I as usual, then the sulphates of lead, barium, strontium and calcium (partly). By boiling the filtrate from this precipitation with 20% sodium thiosulphate, the sulphides of copper, mercuric, bismuth, arsenic, antimony and tin are precipitated.

Defrance²⁶ has also advanced a scheme, outlined in Table VIII, which includes five of the less common elements. Thallium, lead, silver and mercurous are removed by hydrochloric acid; arsenic, bismuth, copper, mercuric, molybdenum, platinum and antimony by sodium thiosulphate. The addition of ammonium chloride and ammonia then removes iron, aluminium, chromium, lead, tin and phosphate. The alkaline earths are then precipitated as carbonates, while the filtrate from

TABLE VIII
To a solution of the cations add HCl



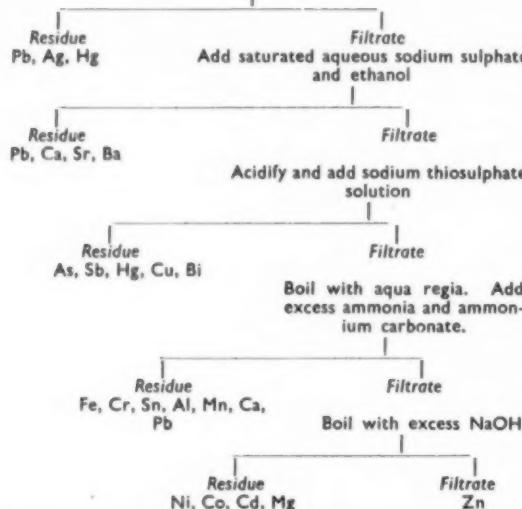
this group, on boiling with sodium hydroxide, precipitates manganese, nickel, cobalt, cadmium and magnesium, leaving zinc, lithium and potassium in solution.

Mee's scheme,²⁴ resembles that of Defrance in the composition of the group precipitates, although the order of the groups is different. In it, and in its small-scale modification, developed by Belcher and Burton,²⁷ and further modified by Belcher and Wilson,²⁸ the cations are divided into five groups, as shown in Table IX. The first group reagent is hydrochloric acid. This, as usual, precipitates the chlorides of silver, mercurous and lead. To the filtrate from this, saturated sodium sulphate solution and a little ethanol are added, and on standing for a short time the sulphates of lead, calcium (partially), strontium and barium are precipitated. The solution is now made feebly acid by first neutralising with ammonium hydroxide, and then adding a few drops of dilute hydrochloric acid, sufficient to make the solution just slightly acid. Metals forming sulphides which are insoluble in dilute acid (arsenic, antimony, mercury, copper and bismuth) are then precipitated by the addition of sodium thiosulphate solution.

The filtrate from this precipitation is boiled with aqua regia to destroy excess sodium thiosulphate. Ammonium hydroxide is then added in excess, followed by ammonium carbonate. This precipitates iron, chromium, tin, aluminium, manganese, and any calcium and lead which may have escaped precipitation earlier. On boiling the filtrate with a considerable excess of

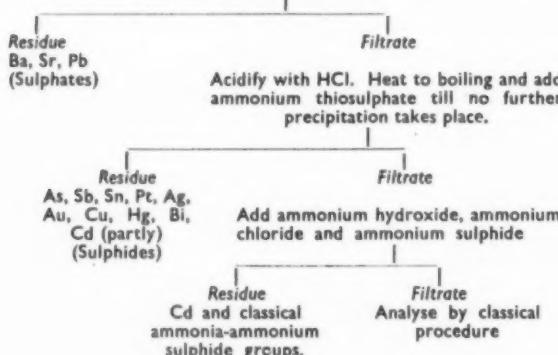
sodium hydroxide, the hydroxides and basic carbonates of nickel, cobalt, cadmium and magnesium are precipitated. The filtrate from this group can only contain zinc, which is precipitated by the action of potassium ferrocyanide. Sodium and potassium are detected in a separate sample of the original substance by special tests. The small scale modification precipitates all the calcium in one group, and also brings down the zinc as sulphide.

TABLE IX
Treat with HCl



Orlowski²⁹ makes a double use of ammonium thiosulphate in a scheme (Table X) in which Group I is removed as usual. The addition of ammonium thiosulphate to the neutral or faintly acid filtrate precipitates the alkaline earths as sulphates. This is the only example of this reaction employed in this way, other workers using sulphuric acid or alkali sulphates for the precipitation. After this treatment the filtrate is acidified, and further addition of the reagent causes the precipitation of the sulphides of arsenic, antimony, tin, platinum, gold, copper, mercury, bismuth and cadmium, the last two incompletely. The filtrate from this procedure is further treated by well-known reactions.

TABLE X
To the filtrate from Group I add ammonium thiosulphate, after making neutral or slightly acid.



Discussion

Of the various reagents considered in this section, thioacetic acid and its ammonium salt gained some popularity at the beginning of the century, but have lost support in the last twenty years. Although excellent results have been claimed for them¹⁸ they are even more objectionable, from a hygienic point of view, than gaseous sulphureted hydrogen, and it has been pointed out by Alvarez²⁰ that the ammonium salt is unstable, and must be prepared as required. From the hygienic point of view, hydrogen sulphide in acetone is also objectionable.

Sodium thiosulphate, on the other hand, is still being used, and Vortmann²⁵ claims that the results obtained with this reagent are equally satisfactory with those obtained using hydrogen sulphide. Cornog⁸ agrees that the separations obtained are clean and complete, provided that the acid concentration is properly adjusted, but he objects to the use of the reagent firstly because of the formation of free sulphur, and secondly because the adjustment of the acidity requires considerable experimental skill, and being, he believes, beyond the abilities of inexperienced workers—in his case, undergraduates.

On hygienic grounds the reagent is much superior to gaseous hydrogen sulphide. Another point worth noting is that in the small-scale modification by Belcher and Burton⁷ regulation of the quantities must, by the method of operation, be more exactly carried out than in most macro procedures, thus removing one of Cornog's objections to a considerable extent.

Apart from the schemes due to Cornog⁸ and Chao and Hwang,⁹ which use pH control, the schemes depending on the use of alkali sulphides show no novel features. The attainment of the correct acidity using alkali sulphides, either by addition to an acid solution or to an alkaline solution followed by acidification, is a difficult operation, and consequently these schemes have not been used to any extent in the precipitation of Group II, although the use of sodium sulphide has been advocated by Richter³¹ instead of an aqueous solution of hydrogen sulphide, on account of its greater stability.

However, the application of the alkali sulphides to other schemes of analysis has been fairly widespread. Though no definite criticisms have been made regarding these schemes, the use of ammonium sulphide and separations in highly alkaline solution has been adversely commented upon^{8, 32}.

Of the other reagents mentioned, thiourea and sodium diethyldithiocarbamate have been considered unsuitable, and none seems to have been investigated to any extent.

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METALLURGICAL DIGEST

Where Does Powder Metallurgy Stand To-day?

By H. R. Clauser

THE progress of powder metallurgy as a large scale production process is closely tied to advances made in metal powders, and to the availability of high quality metal powders at reasonably low cost. The past few years have seen improvements in both the processing characteristics and properties of metal powders, and development of a number of new powders with higher mechanical properties. But there has been no noticeable downward trend in the cost of metal powders.

The principal metal powders presently used in powder metallurgy are copper, bronze, brass and iron. Of these, bronze is used in the greatest quantity, its chief application being for self-lubricating porous bearings. Brass and iron powders are the most widely used for mechanical parts. Brass is in a more favourable position, when the cost of brass powder is compared to that of brass wrought stock and of brass ingots. In the case of iron, the difference is much larger. The expansion of the use of iron powder is closely linked with the development of lower cost high quality powders.

The relatively low mechanical properties, such as strength and ductility, compared to those obtainable in cast and wrought metals has always been and still is an important factor upon which the growth of the use of metal powder parts depends. Development of pre-alloyed powders has been an important step towards improving the properties of metal powder parts. In pre-alloyed powders two or more elements are present in the powder in alloyed form; thus, the alloying action does not depend upon diffusion during the sintering operation.

There are several advantages obtained with pre-alloyed powders. Blending and mixing operations are eliminated; the powders have more uniform processing characteristics and

provide a more uniform product; the sintering time is reduced over that necessary for diffusion sintering; and perhaps most important, mechanical properties are improved.

While pre-alloyed steels are available in the range from 0.20 to around 1.20% combined carbon, the higher carbon contents find only limited use, because high particle hardness considerably reduces their compressibility.

Brass powders have been pre-alloyed commercially for a number of years and their use for mechanical parts has been expanding. Considerable success has been reported for the process in which iron powder parts are impregnated with brass or lead during the sintering operation to increase density, strength and ductility.

Aluminium powder is also beginning to receive some attention for use in making small parts. A new aluminium powder is reported to have properties close to those of wrought pure aluminium in the annealed condition. In this powder the oxide film on the particles is removed by an etching agent which then serves as the lubricant during pressing.

The future of powder metallurgy as a production process depends in no small measure on how much is known about the variables, and how well they can be controlled. In recent years considerable strides have been made in controlling these variables. The uniformity and purity of powders has been improved so that the fabricator knows reasonably well what he is getting. However, the study of all the processing variables has really only begun. Relatively little is known about the effects that all the variables, encountered during pressing and sintering, have on final properties of various metal powders and on dimensional changes in the finished part. More basic investigations of the sintering mechanism are particularly needed to provide data on the differ-

ences in sintering characteristics for different materials, and to determine the effects of critical small changes in time, temperature and atmosphere.

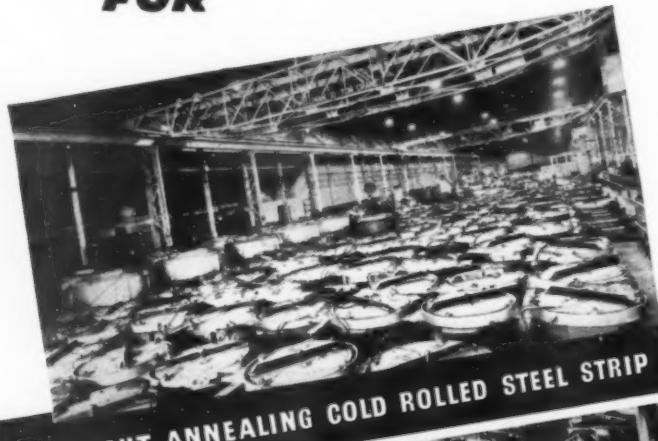
Equipment manufacturers, until recently have done little to provide equipment specifically designed to meet the special requirements of powder metallurgy. The demand is for presses with higher pressures and faster production. Unfortunately, these two demands cannot always be satisfied. For high production rates, mechanical type presses are generally used, but where very high pressures are involved it is necessary to use hydraulic types, which are slower in speed. Thus, the problem of the press manufacturer is to reconcile these differences.

Sintering furnaces also can be improved upon. There are a large number of variables in the sintering operation, and furnaces must be designed giving due consideration to these variables.

Much has been heard during the past few years about hot-pressing. Experiments have shown that when metal powders are pressed in a hot instead of cold condition, extremely high density parts are possible. Also, the pressing pressures required are much lower than those required for cold-pressing. Hot-pressing would also aid the expansion of the use of alloy powder parts. While hot-pressing is being used successfully for making parts of high temperature powders such as tungsten carbide, it is still in the development stage for the other common powders. Before it can be used extensively on a commercial basis a number of problems must be solved. These include the prevention of oxidation during heating, elimination of excessive die wear, and working out means of rapidly heating dies and punches.

In recent years the possibilities of plating metal powder compacts has received considerable attention. Although it has been found that most metals which can be plated in wrought form can also be plated when in the form of a metal powder compact, the results are by no means comparable. There are inherent difficulties which still remain to be worked out before

G.E.C. ELECTRIC FURNACES FOR



BRIGHT ANNEALING COLD ROLLED STEEL STRIP

Photo.: The Whitehead
Iron and Steel Co.,
Ltd., Newport, Mon.

G.E.C. experts are
available at all times
to advise on all types of
Process Heating
equipment. Technical
information will be
gladly supplied on request



HEAT TREATMENT OF LIGHT ALLOYS

Photo.: The British
Aluminium Co., Ltd.,
Falkirk.



BILLET HEATING for EXTRUSION PURPOSES

Photo.: Earle, Bourne
and Co., Ltd., Birming-
ham.

THE GENERAL ELECTRIC CO., LTD. MAGNET HOUSE, KINGSWAY, LONDON, W.C.2

metal powder parts can be plated as satisfactorily as the wrought forms.

Progress has been made in other directions—for example, heat treating. Ferrous powder parts are being successfully heat treated and case hardened to improve their strength and wear properties. And impregnation processes are being used to raise the performance characteristics of metal parts.

Powder metallurgy has about reached the point where closer co-operation among powder producers, fabricators and users is essential to further expansion, and there are a number of signs that indicate progress in this direction. Another indication is the increased activity in

establishing standards and specifications, as pointed out earlier in this article. Finally, there is the problem of proper application of the process. Powder metallurgy has suffered probably more from over-application than from under-application. Its limitations are now better known and the tendency is to use powder metallurgy only in applications where satisfactory performance is assured. However, there are some who feel that much more could be done to eliminate misapplications. There is no doubt that the sooner the capabilities and limitations of powder metallurgy are more clearly defined and generally known the sooner it will find its rightful place in industrial production.

New Slab Heating Furnace

A NEW slab heating furnace, with a maximum heating capacity of 105 tons per hour, has been installed at the Irvin Works of the Carnegie-Illinois Steel Corporation to provide additional slab heating facilities for the 80 in. hot strip mill. It is zone controlled, triple fired, end charged, and discharged, and continuous, and includes a recuperator, employing silicon-carbide type tubes, which is of sufficient size to preheat all combustion air to about 800° F. It is designed to handle slabs from 3-8 in. thick, 20-60 in. wide, and 60-216 in. long.

Primary heating zone of the furnace has separately controlled over-and-under-fired burners, while the secondary soaking zone has over-fired burners. Fuel normally used is coke oven gas; however, the furnace was designed to permit the use of oil in the event of gas failure. The gas burners are of the controlled, long-flame luminous type. The waste-gas flue is arranged so that the products of combustion can be discharged to either the stack or to the collecting flue for the waste heat boilers.

A silicon-carbide type brick and alloy steel dry-skid construction was used for the hearth and slag zone. Roof of the furnace is of suspended high-temperature ceramic-type tile. Silicon-carbide type brick were used in the walls from the bottom to 1 ft. above the skid line. High-temperature quality insulating refractory brick were used from 1 ft. above the skid line to the suspended roof.

Inspection doors of the furnace are of cast iron, refractory lined and operated by air cylinders. Slag doors, bricked up when the furnace is in

operation, are approximately 2 ft. wide by 30 in. high to the spring line of the arch to permit access. The double, cast iron, refractory-lined charging doors are operated by an electric thruster. A water-cooled lintel is provided at the charging doors. The double, cast iron, refractory-lined discharge doors are operated automatically by electric motors. Both charge and discharge doors are interlocked with the pusher controls.

Four main water-cooled skids were provided, with two skids located on each side of the furnace centreline. These skids were constructed of double extra heavy oil still tubing of the 4-6% Cr type. Wearing bars were welded continuously along the top surface. Skids in the main heating zone are carried on five water-cooled cross supports braced to resist transverse stresses. Provision was made to accommodate expansion and contraction of all the skid pipes and supports.

The waste-gas flue, including the self-supporting steel stack, is lined with 6 in. of second quality firebrick, with all necessary insulation. The flue was arranged so that waste gas could be discharged to the stack or to the waste-gas collecting flue. Particular attention was paid to elimination of high draft loss at intersections of flues, such as on the back of the furnace recuperator, or stack. A main-control damper operated from a furnace pressure controller was provided in the single flue section. The damper was constructed of 25-12 chromium nickel, alloy sheet of double corrugated design with a cast frame. A high-chrome alloy, heat resisting, mushroom-type, shut-off damper was provided in the existing waste-heat-boiler collecting

flue. The mushroom seat casting was installed on a cast monolithic base to resist shock incident to operation of the valve. A water-cooled stack damper was provided, studded to retain a covering of plastic refractory cement. The waste-gas flue damper and stack damper are individually operated by oil hydraulic pistons, located at the dampers and controlled by hand valves located in the waste-heat boiler house. A 2½ in. self-piloted, inspirator-type coke-oven gas burner was installed at the base of the stack.

In operation, the gas flow to the top and bottom burner sections of the main heating zone is operated manually from the furnace panel or automatically by temperature controllers. The gas flow to the soaking zone is operated automatically by temperature controllers or manually through a push button on the panel. The air flow to each zone is controlled automatically or manually by gas-flow: air-flow ratio controllers located on the panel.

Sintered Iron-Carbon Materials

By H. WEIMER and W. A. FISHER

ALTHOUGH it is known that production variables have a considerable effect on the properties of iron and carbon sintered parts, relatively little fundamental work has been done in its determination. A useful contribution to knowledge in the field is given in this report of an investigation on the influence of sintering time, temperature and atmosphere, as well as the type of powder and carbon addition.

In vacuum sintering, it was possible to obtain ferritic-graphitic, ferritic-graphitic-pearlitic or pearlitic structures. Changes in the types of graphite and iron powders affected the sintering temperature at which each structure was obtained. When the raw materials and sintering conditions were constant, these structures were reproducible. The tensile strength varied from about 1-38 tons/sq. in., depending on the amount and type of the carbon addition, the sintering time and temperature.

The atmosphere during sintering had no noticeable effect on the mechanical properties of the sintered parts. Hydrogen caused a greater carbon loss than nitrogen or vacuum, while carbon monoxide was satisfactory if the specimen was heated rapidly through the lower temperature ranges. Under these conditions, sintering in solid carbonaceous media resulted in an increase in carbon of about 0.8%;

this absorbed carbon diffused uniformly throughout the specimens.

The inter-relationship of hardness, tensile strength and carbon content (0.3-1.0%) was determined for samples compacted under 42.5-57 tons/sq. in. and sintered for 2-8 hours at 1,200° and 1,245° C. There was a

linear relationship between hardness and strength. For each compacting pressure, there was also a linear relationship between the final carbon content and the hardness, with the higher pressures giving higher hardness for a specific carbon content. There was more scatter in the relationship

between tensile strength and elongation, but the higher pressures tended to give higher elongations.

A few tests were made on heat-treated sintered samples. Although tensile strengths over 63 tons/sq. in. could be obtained, the specimens were very brittle.

Flame Spraying Steel on Aluminium

A GROUP of research studies was initiated in 1944 by the L'Aluminium Français, la Société Générale du Magnesium and La Société Nouvelle de Métallisation with the object of studying the possibilities offered by the projection spraying of steel on the light and ultra-light alloys. A committee was appointed, and after a considerable number of meetings and a good deal of experimental work, the group agreed on the following practical conclusions. The flame projection of steels of different qualities, from soft mild steels up to steels with a high carbon content, as well as the stainless steels of the 18/8 type or containing 13% of chromium, is possible on the light and ultra-light alloys. The adherence obtained is not the result of an alloy, but of a mechanical keying, with auto-frettage in the case of revolving components with a convex surface, such as tubes and shafts. In the case of concave components, besides this mechanical preparation, a procedure of preheating of the support member of the sprayed coating is necessary, in such a manner that a keying of the deposited sprayed metal is obtained.

The principal characteristics of the deposits are as follows:-

Hardness.—The deposited metal is harder than the initial steel used for the flame spraying by virtue of the rapid cooling effect obtained, and as a result of the work-hardening effect obtained by the crushing effect due to the impact speed of the metal particles.

Frictional Properties.—By virtue of a definite porosity, the deposited coating evinces interesting properties of auto-lubrication and anti-frictional effect. The coefficient of friction is below that of the same steel in the rolled form.

Corrosion Resistance.—In spite of this porosity, and starting with a certain thickness of deposited metal (1 mm.) one can regard the coating as impermeable and that the steel-light alloy couple need give rise to no apprehension. When the light or ultra-light metal is only locally covered

the couple can be neutralised by a band of sprayed zinc.

Machinability. The sprayed mild steels can be machined with high speed steel tools; the cutting angles and the cutting speed should be the same as those used in machining cast iron. Steels with 0.6% carbon can be machined with sintered carbide cutting tools

Finally, it can be said that this method of conferring to light and ultra-light alloys, allows a wear resistance comparable to that of steel. This new technique is particularly interesting in the cases where it is necessary to lighten the weight of a component piece and in such cases where aluminium or magnesium could not be employed, because of lack of sufficient wear resistance.

Cycle Annealing in Quality Steel Production

By Erik Tholander

THE problem of making use of isothermal transformation of austenite after the hot working of steel has been studied by a committee of the Technical Research Board of Jernkontoret. Initially, small samples of four different steels were treated on a laboratory scale in order to investigate the influence of different factors on the results obtainable. These steels, noted in Table I, were chosen as representative of ranges of air- or oil-hardening alloy steels in common use in Sweden.

TABLE I.—COMPOSITION OF SAMPLES

Type	C	Cr	Ni	Mo
Ni-Cr Constructional Steel	0.35	1.2	2.5	—
Ni-Cr-Mo	0.5	0.8	3.0	0.3
Low Alloy Steel	1.0	1.5	—	—
Stainless Steel	0.3	14.0	—	—

Further investigations were made on billets both in laboratory scale and technical production scale. In some cases billets were used, which had been prepared by boring longitudinal or transversal holes and placing small test pieces in the holes as described in the article. Thus, a correlation between the results obtained with small samples and those with billets could be made.

Data for isothermal transformation to pearlite have been obtained by experiments with small samples, for a great number of alloy steels newly standardised in Sweden, the results of which are given in the paper. For all the steels investigated the temperature

range for transformation to pearlite is identical with one or more of four ranges: Group I, 760°-740°; group II, 710°-690°; group III, 670°-650°; and group IV, 625°-600°. Only two of these steels could not be transformed to hundred per cent.

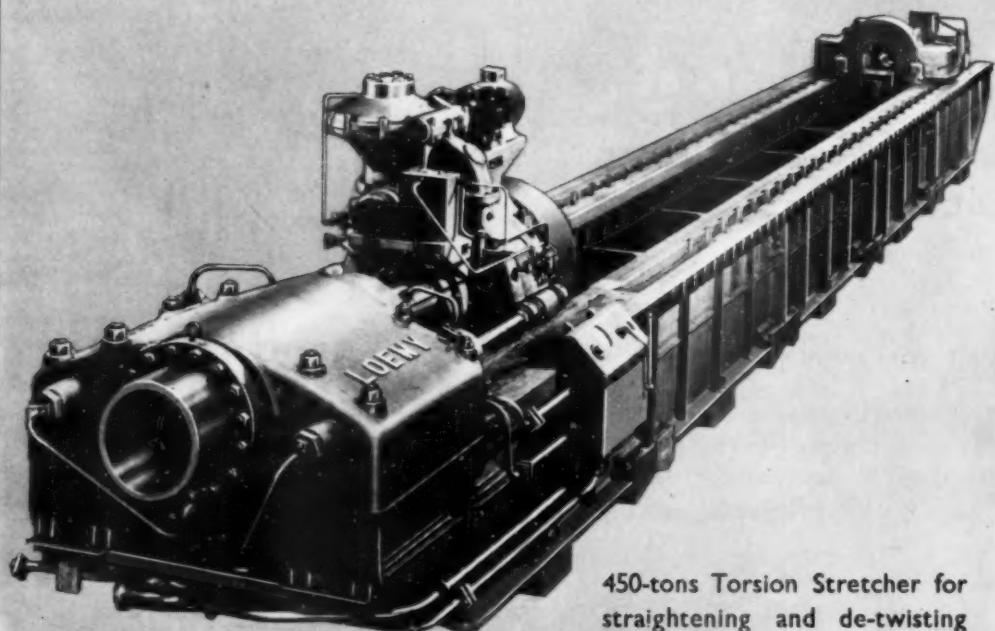
Segregation retards the transformation in ingot and large billet section structures but has no great influence on the transformation in small bar sections. Machinability tests showed good results for pearlitic structures but bad results for bainitic ones. The hardness of bainitic structures was then much higher than that of pearlite.

The conclusion of the investigation is that isothermal transformation to pearlitic structure with good machinability is possible in short time for most of the common alloy steels of oil or air hardening types. Advantages such as elimination of cracks (but not flakes) by cooling, pickling or grinding, better machinability, elimination of separate annealing operations after cooling etc. can be obtained by cycle annealing in connection with a previous hot working operation. However, the committee has not investigated the problem of suitable furnaces.

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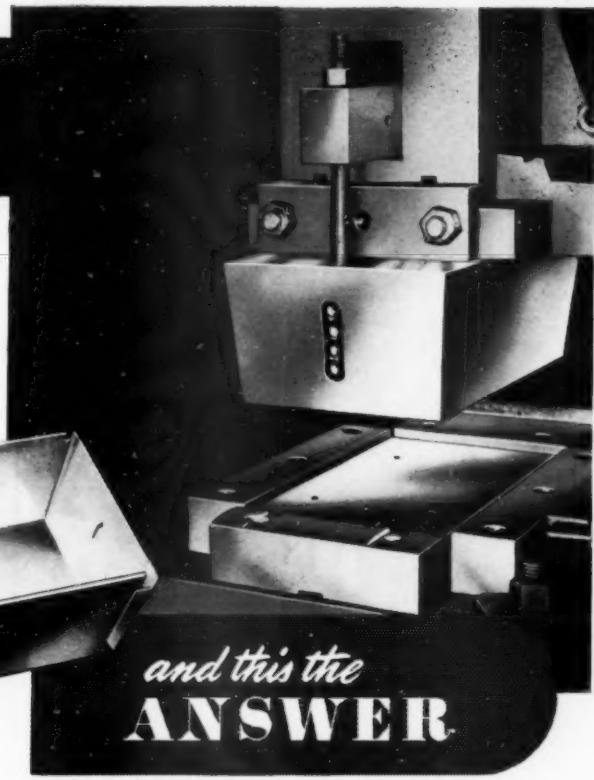
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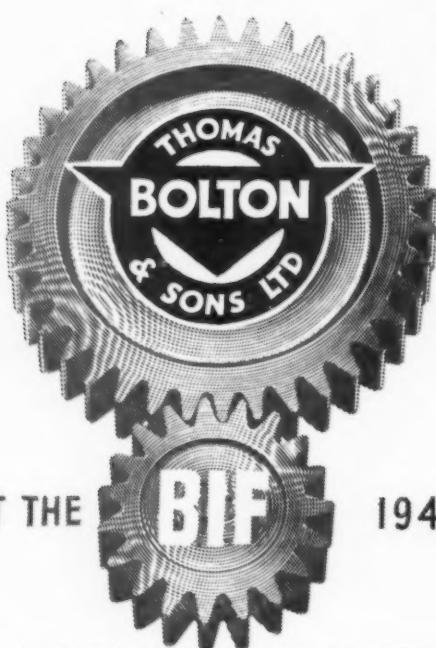
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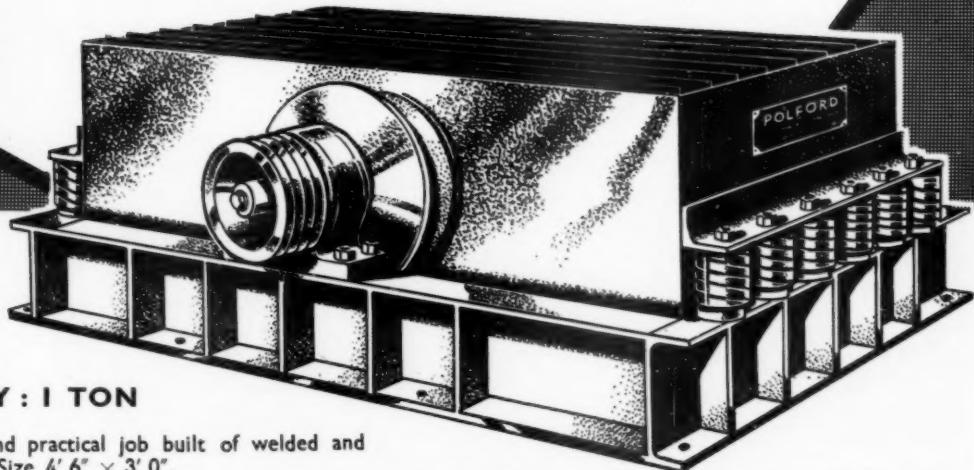
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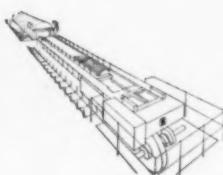
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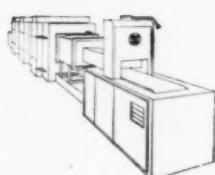
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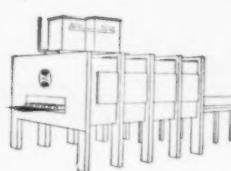
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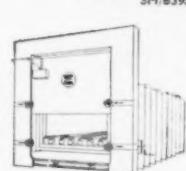
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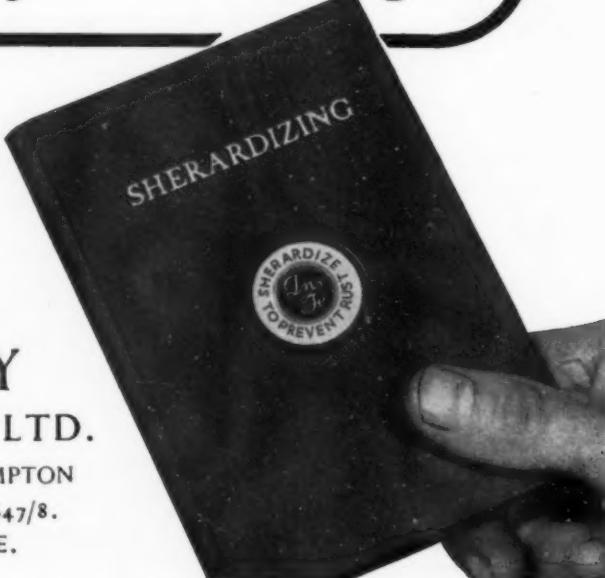
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MISCELLANEOUS ADVERTISEMENTS

SITUATIONS VACANT

Vacancies advertised are restricted to persons or employments excepted from the provisions of the Control of Engagements Order, 1947.

APPLICATIONS are invited by the Standard Telecommunication Laboratories, Ltd. for a Metallurgist or Chemist to assist with a programme of preparation and testing of specialised materials. Applicants should preferably have a University Degree, but real interest in experimental work is more important than academic qualifications. Salary in accordance with qualifications and experience. Applications should be made to the Personnel Manager, Standard Telecommunication Laboratories, Ltd., Progress Way, Enfield, Middlesex.

LEADING Sheffield manufacturer of engineers' small tools requires *First Class Metallurgist* to take charge of all heat-treatment operations. Salary £1,000 per annum. Only thoroughly competent persons with a first rate background in H.S. and carbon steel heat treatment need apply. Ability to control a labour force of upwards of 50 persons and be completely responsible for the results a necessity. Acceptance and testing of raw materials also comes into the position. Apply in confidence in first instance to Box No. MD.10, METALLURGIA, 31, King Street, West Manchester, 3.

SALES Manager/Representative wanted with experience in handling ferro-alloys. Should have contacts in Sheffield area or in export business. Please state age, experience and salary or commission required. Box No. MD.12, METALLURGIA, 31, King Street West, Manchester, 3.

TWO qualified chemists required for the laboratory of a light engineering firm in the South West, for metallurgical and miscellaneous analyses and investigations of a non-routine character, and to carry out a development programme on protective treatments of light alloys. Applicants must have a recognised qualification, and previous experience in a works laboratory would be advantageous. Both positions are permanent and offer scope for advancement. Apply stating age, experience and salary required to Box No. MC.4, METALLURGIA, 31, King Street West, Manchester, 3.

NATIONAL FOUNDRY COLLEGE

THE Governors invite applications for post of Lecturer in Foundry Technology. Candidates must have had sound industrial experience in the foundry industry. Experience in pattern making or works control an advantage. University degree or equivalent is desirable. Salary £750 per annum, rising to £850 per annum by annual increments of £25.

Full particulars from the Clerk to the Governors, National Foundry College, Education Offices, North Street, Wolverhampton.

NATIONAL FOUNDRY COLLEGE DIPLOMA COURSE OPENS IN SEPTEMBER, 1949

Students seeking admission should make application without delay. Full particulars from the Head of the National Foundry College, Wulfruna Street, Wolverhampton.

Metallurgia Monographs : No. 1

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SITUATIONS WANTED

ELECTRICAL Engineer, 14 years' practical experience of physical metallurgy and application of metals to light engineering, with business experience, requires responsible executive or technical sales position, Box No. MC.8, METALLURGIA, 31, King Street West, Manchester, 3.

PUBLIC APPOINTMENTS

MINISTRY OF SUPPLY invites applications for unestablished appointments in the following grades for work on the physics of metals in Research and Development Establishments in London and South England:—

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For the Scientific Officer grades a good honours degree in metallurgy, physics or chemistry is required. Appointment in these grades carries F.S.S.U. benefits.

For the Experimental Officer grades the minimum qualification is Higher School Certificate or Higher National Certificate in chemistry or equivalent qualification in metallurgy.

Application forms obtainable from Technical and Scientific Register (K), York House, Kingsway, London, W.C. 2, quoting F.1190/48A. Closing date 21 days from the date of this publication.

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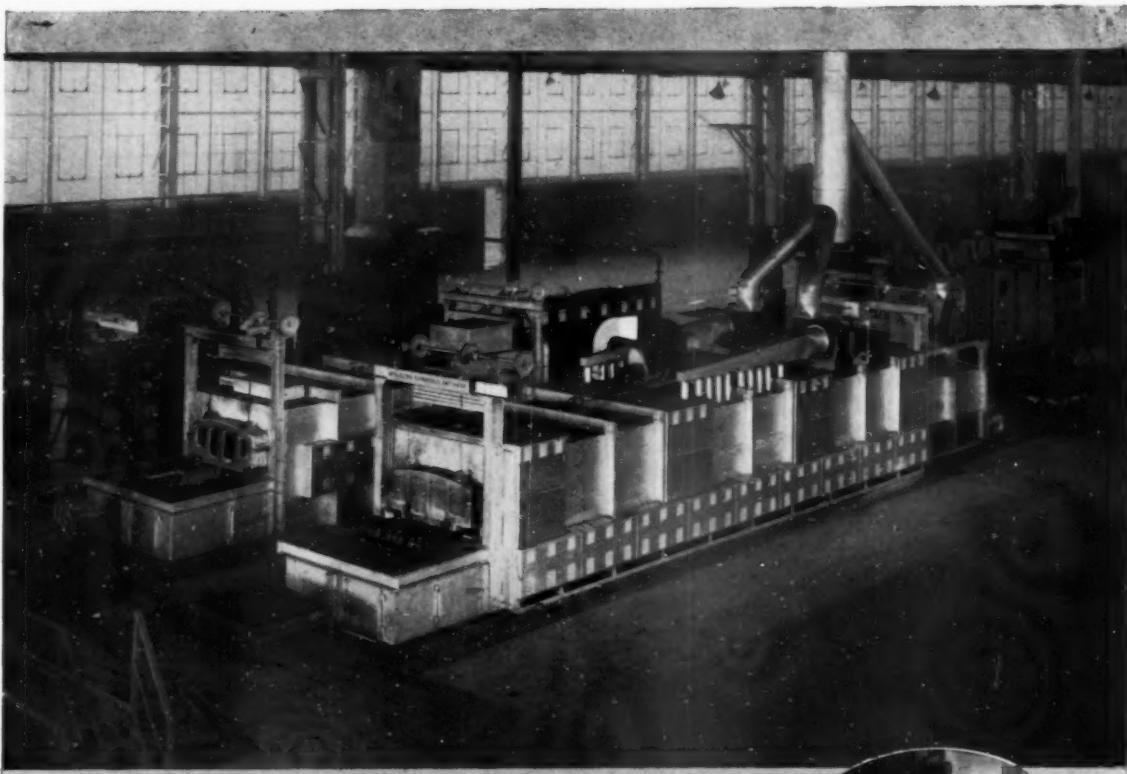
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